

Exhibit 26

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PHASE REPORT

on

**AN INVESTIGATION OF THE MEASUREMENT
AND CAUSATIVE FACTORS OF LUSTER
IN FINE PARTICULATE TALCS**

to

JOHNSON AND JOHNSON

September 15, 1959

by

James F. Shea, Charles B. Sclar, and H. B. Kinnear

**BATTELLE MEMORIAL INSTITUTE
505 King Avenue
Columbus 1, Ohio**

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Battelle Memorial Institute

505 KING AVENUE COLUMBUS 1, OHIO

September 24, 1959

Mr. W. H. Ashton
Research Department
Johnson and Johnson
New Brunswick, New Jersey

Dear Mr. Ashton:

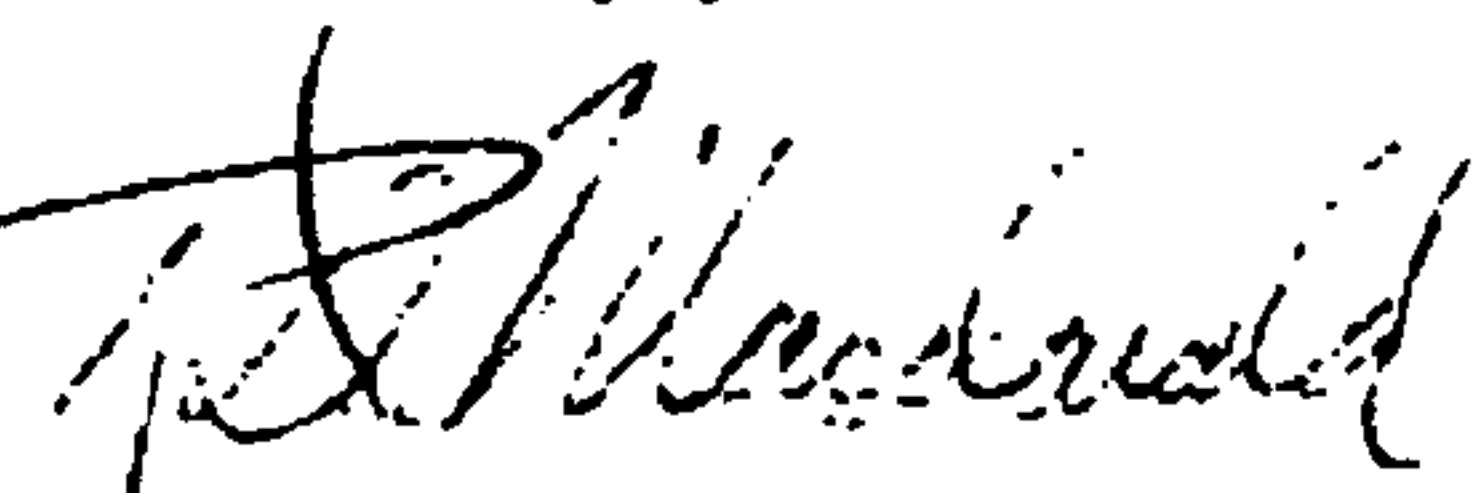
We are sending you six copies of our report "An Investigation of the Measurement and Causative Factors of Luster in Fine Particulate Talc's" by James F. Shea, Charles B. Sclar, and H. B. Kinnear.

We believe this investigation to be an important one because, although it was not an exhaustive study, strong evidence is presented that talc should be wet ground to obtain maximum luster. The study shows further that there are logical reasons why this should be true, reasons which are connected with the crumpling and deformation of talc platelets during grinding, with surface roughness, and with platelet diameter-to-thickness ratios.

The correlation of luster with method of grinding would not have been possible without the development of a positive technique for the measurement of the luster of fine particulate talc. Here again, no attempt was made to develop a highly refined method. The method as described is, however, very satisfactory for the present needs of Johnson and Johnson.

We shall welcome your comments on this report.

Sincerely yours,



R. D. Macdonald
Assistant Chief
Minerals Beneficiation Division

RDM:ims
Enc. (6)

DEDICATED TO THE ADVANCEMENT OF SCIENCE

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TABLE OF CONTENTS

	<u>Page</u>
SUMMARY	1
INTRODUCTION	3
DEFINITION OF LUSTER	3
PRELIMINARY WORK TO DEVELOP A SUITABLE METHOD FOR MEASURING LUSTER.	5
Preparation of a Set of Visually Rated Luster Standards	5
Physical Measurement of the Luster Standards	5
Specular Gloss	7
Reflectance	7
Whiteness and yellowness	7
Contrast Gloss of En Masse Samples	7
Contrast Gloss of Thin, Oriented Talc Surfaces	8
PROCEDURE DEVELOPED FOR MEASURING THE CONTRAST GLOSS OF TALC SAMPLES	8
Precision of the Contrast-Gloss Measurement	9
Accuracy of the Contrast-Gloss Measurement	11
Possibility of Refining the Method	12
CONTRAST-GLOSS MEASUREMENTS ON A SUITE OF TALC PRODUCTS FROM THE LABORATORY DEVELOPMENT WORK.	13
INSTRUMENT VERSUS EYE IN CONTRAST-GLOSS RATING	16
EVALUATION OF THE METHOD	17
A STUDY OF THE CAUSATIVE FACTORS OF LUSTER	17
The Effect of Chemical Differences in the Talc and Physical Environmental Factors	18
Loss on Ignition	18
Acid-Soluble Impurities	21
Total Impurities	21
Sensible Moisture	22
Possibility of Loss of Luster Over a Period of Time	22
Effect of Particle Size	22
Effect of the Method of Grinding	24
Effect of Morphology and Dimensions of the Talc Particles	25
Samples Investigated	26
Procedure	27
Results	28
Conclusions as to the Effect of Morphology and Dimensions of Talc on Luster	31
GENERAL CONCLUSIONS	32

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J&J-0082494

TABLE OF CONTENTS
(Continued)

	<u>Page</u>
APPENDIX A	
ITEM 1. DESCRIPTION AND INSTRUCTIONS. THE GARDNER MULTIPURPOSE REFLECTOMETER	
ITEM 2. NATIONAL BUREAU OF STANDARDS RESEARCH PAPER 1345	
APPENDIX B	
PRELIMINARY EXPERIMENTS TO RELATE OPTICAL MEASUREMENTS TO VISUALLY RATED LUSTER	B-1
APPENDIX C	
THE RELATIONSHIP OF THE LUSTER OF GROUND PLATY TALC TO THE MORPHOLOGY AND DIMENSIONS OF THE TALC PARTICLES	C-1
APPENDIX D	
PHOTOMICROGRAPHS OF CROSS SECTIONS OF TALC PLATELETS IN FLOTATION CONCENTRATES AS OBSERVED IN INCIDENT LIGHT IN POLISHED RESIN MOUNTS	D-1
APPENDIX E	
STANDARDS FOR CONTRAST-GLOSS MEASUREMENT	E-1

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AN INVESTIGATION OF THE MEASUREMENT AND CAUSATIVE FACTORS OF LUSTER IN FINE PARTICULATE TALCS

by

James F. Shea, Charles B. Sclar, and H. B. Kinnear

SUMMARY

During the laboratory development work on the beneficiation of Italian talcs to produce a platy-talc concentrate it was continually observed that the flotation concentrates, the finished product, appeared to be considerably more "lustrous" than the unbeneficiated talcs themselves and much more "lustrous" than the products rejected by the beneficiation procedure. Insofar as the eye could perceive, the flotation products made from wet-ground run-of-mine talc appeared more lustrous than those made from the Italian No. 2 talc which had previously been dry ground in a Raymond-type mill in Italy.

In the belief that "luster" might be a valuable property and that the wet-grinding process which is a part of the Battelle-developed process might be providing an extra dividend in the way of enhanced luster, a brief research program was undertaken to determine:

- (1) Whether the phenomenon of luster, which had previously been rated visually only, could be measured instrumentally
- (2) If a satisfactory method for measuring luster could be found, whether the physical measurements would bear out the conclusions derived from visual observation, viz., that wet-grinding enhanced luster
- (3) The effect of certain chemical and physical, intrinsic and environmental factors on the luster of talc and talc products.

A survey of several methods of optical measurement indicated that the measurement of the contrast gloss of talc samples rubbed onto analytical-grade filter papers yielded numerical results which coincided well with those of visual observation. The procedure for conducting this determination, the description of the equipment, and the significance of the phenomenon of contrast gloss are dealt with in detail in this report. In brief, contrast gloss is an empirical value and is a measure of the ratio of the amount of light specularly reflected by a sample to the amount of light diffusely reflected at the same time from the same area of the sample.

Although the method tentatively prescribed lacks some refinement, it is, in the hands of a practiced operator, sufficiently precise to serve as a means for inspecting the luster of talc products.

The method was applied to a suite of samples prepared during the laboratory development work. The more significant results were:

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- (1) The contrast gloss, i.e., luster, of flotation concentrates, was considerably higher than the contrast gloss of unseparated material.
- (2) The contrast gloss of flotation concentrates made from wet-ground run-of-mine talc was superior to that of concentrates made from Italian No. 2 talc which had previously been ground to minus 200 mesh in Raymond-type mills in Italy.
- (3) The contrast gloss of flotation concentrates made from wet-ground run-of-mine talc was generally superior, although there was an exception, to that of concentrates made from the run-of-mine talc which was dry ground in a pebble mill at Battelle.
- (4) The contrast gloss of run-of-mine talc feed material and its various products excelled the contrast gloss of corresponding Italian No. 2 talc materials.

These data confirm the judgment, previously based on visual inspection, that the beneficiation process upgrades the talc significantly with respect to luster. They also point strongly to the probability that wet grinding enhances luster. This latter conclusion is not clear cut because the inferior luster of the Italian No. 2 products may be due, at least in part, to an intrinsic property of this talc; or to the possibility that, having been ground months ago, it lost luster over a period of time. Furthermore, the data on the run-of-mine talc dry ground in the pebble mill are too limited for a decisive conclusion.

An investigation into the causative factors for luster was carried through preliminary stages. This study indicated:

- (1) The most important determinant of luster probably resides in the morphology or surface characteristics and dimensions of the talc platelets. The combination of factors which seems to accompany high luster is:
 - (a) A maximum amount of platelets with level and regular surfaces
 - (b) Platelets with the largest individual areas
 - (c) Platelets with the highest ratio of area to thickness.

These factors are, of course, a function of the method of grinding. It may therefore be said that the method of grinding can be an important cause of differences in luster.

- (2) While the above factors may represent the sole causes of luster, there is a possibility, based on limited data, that luster may also be determined to some extent by chemical composition and aging.

No additional work on the luster problem is contemplated at this time.

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improvement in beneficiation procedures and to determine the correlative relationships of the physical properties of talc, it is necessary to be able to measure small differences in the physical properties and to be able to compare them to other quantitative measurements. Knowledge of these interrelationships serves as the basis for interpretation of improvement in quality and thus serves to make it possible to visualize methods of beneficiation.

Since the subjective tests are of little help in measuring small differences in one of the many physical properties encountered, and since such tests have no basis for correlation, a machine was built to measure objectively, or test for, abrasiveness, apart from other physical properties.

The Abrasion Machine

Because it was necessary to measure small differences in the abrasiveness of talc, a machine was built to test the wear effect of small concentrations of grit on standard material. The machine was built of a 1/20-hp 1725-rpm electric motor mounted vertically and fitted with a 5-inch lap covered by a Buehler Microcloth held in place by a rubber belt. The lap portion of the machine is set into a steel bowl and covered with a plastic lid. Mounted on a ringstand over the lap a 500-ml open separatory funnel with stopcock is connected by a rubber tube with an adjustable pinch clamp to a feed spout. The separatory funnel contains the sample of talc to be tested in a slurry of 3 grams of talc to 350 ml of water. The feed spout and a cylindrical pellet holder are mounted in a removable crossbar over the lap. Accessibility to these parts is afforded through a hole in the plastic cover. Standard 1/2-inch-diameter pellets are held in the sample holder by a 16.1-gram weight to prevent their skipping or floating on the lap. A 1000-ml beaker mounted under a drain in the steel bowl catches the tested slurry. Figure 1 shows the over-all apparatus. Figure 2 shows the detail of the feed and abrasion mechanism. A detailed description of the abrasion machine and the technique of its operation are found in Appendix A.

In order to measure the abrasiveness of the talc in the slurry a test had to be designed where the object abraded would have a great enough loss to be measured physically. Since the abrasiveness to be measured was that of a powder containing generally from only 1 to 3 per cent of abrasive gangue particles, the material to be abraded had to have a hardness greater than that of the talc, less than that of the grit, and also had to be coherent and homogeneous. After testing a large number of materials it was decided to perform the bulk of the tests on pellets made of minus 400-mesh Italian talc pressed under 50,000-psi pressure. The pellets average 5.20 grams and have dimensions of 1/2 by 7/10 inch. The pellets have a hardness greater than that of the raw talc and less than that of the contaminants (Table 4). Carbonate pellets were made to test specifically for the rarer, harder components, in a similar manner, but using alcohol instead of water in the slurry.

TABLE 4. RELATIVE HARDNESS OF THE TEST PELLETS AND THE GRIT PRESENT IN ITALIAN TALC

Mineral	Moh Hardness
Talc	1
Pressed-talc test pellet	± 2 (scratches talc)
Magnesite	3.5 (scratches talc pellet)
Dolomite	4
Pressed-carbonate test pellet	> 4 (scratches dolomite)
Apatite	5 (scratches carbonate pellet)
Titanite	5
Tremolite	6
Rutile	6
Zircon	7.5

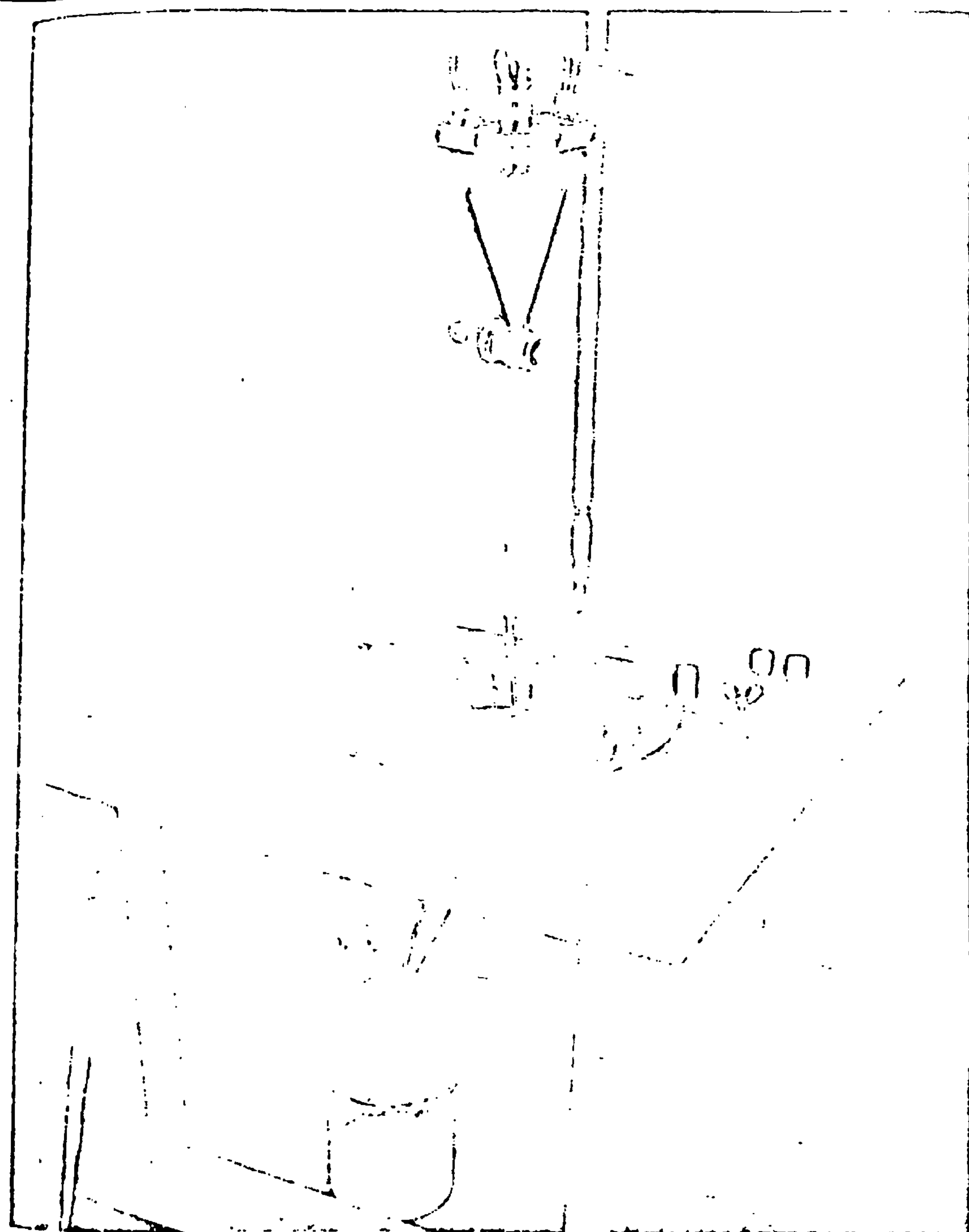
The abrasion-machine operation is timed electrically. The pellets are measured on a micrometer caliper before the test and afterward, after drying. The abrasion measurement is reported in decimal fractions of an inch per second. Although there are limitations to the use of a micrometer, the samples which were compared demonstrated differences in measurement large enough to be significant. Measurements based on weight were found to be entirely unsatisfactory inasmuch as some weight loss was due to spalling and abrasion of the pellet by the walls of the sample tube on portions other than that exposed to the lap and abrasive. This indicated losses which were no indication of the degree of loss due to action on the tested surface alone.

The abrasion machine subjects the standard pellet to abrasion by the sample of talc being studied, at a high rate of speed. It has been calculated that the pellet receives wear equivalent to being rubbed over more than 1800 ft/min of surface of the talc being tested. As expected, the abrasion machine demonstrates that the slurry samples with the greater incidence of mineral contaminants produce the greater amount of abrasion on the pressed pellets. It is also shown that those samples with primarily platy habit are less abrasive than those containing effective amounts of nonplaty talc.

More precise abrasion machines could be built; however, the device used is satisfactory for the purpose of comparing samples within the range of those tested and is an adequate means of obtaining comparable measurements of the effect of grit. Typical figures obtained by the abrasion experiments are shown in Table 5.

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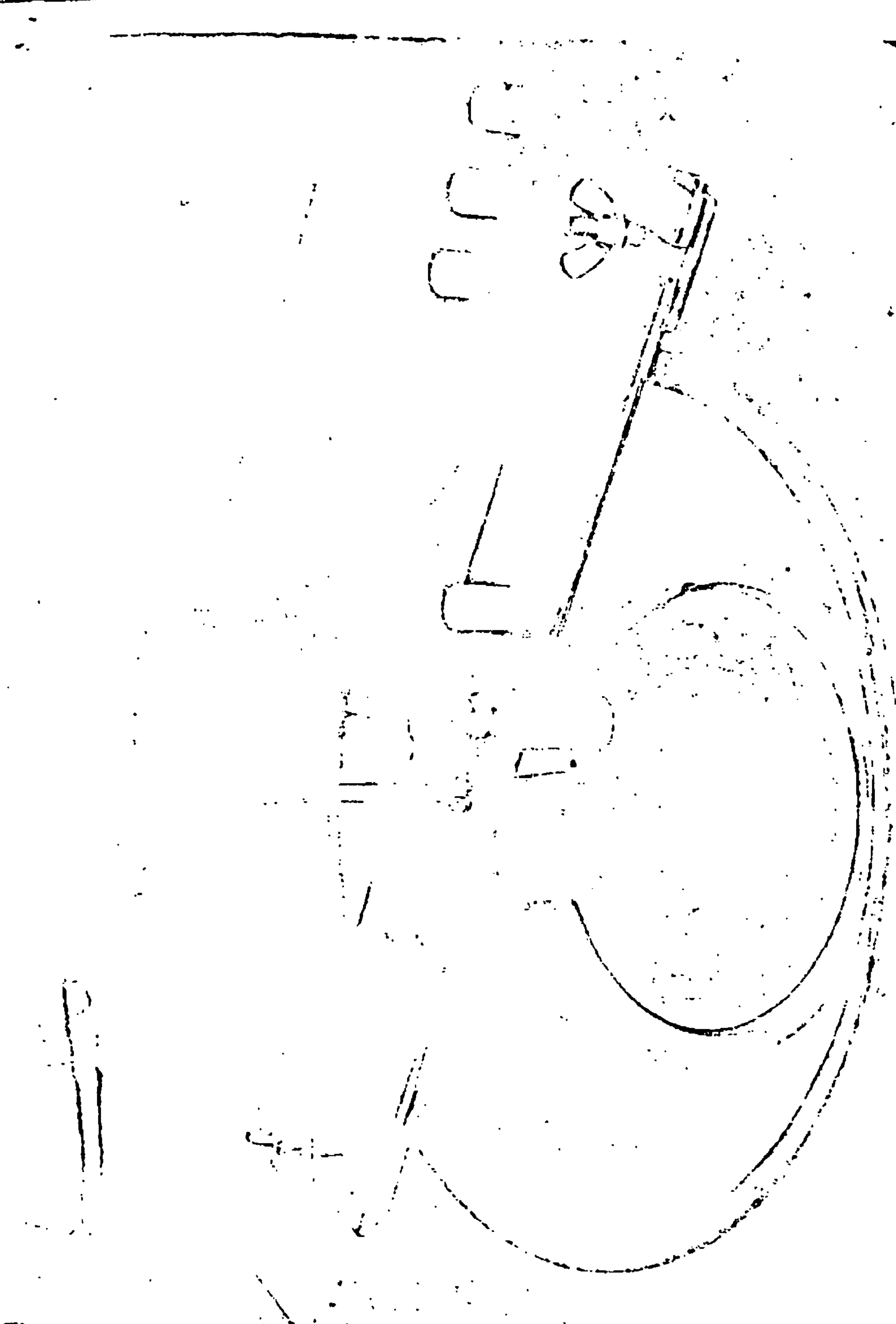


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FIGURE 1. THE ABRASION MACHINE SHOWING RESERVOIR CONTAINING SAMPLE IN SLURRY TO BE TESTED FOR ITS ABRASIVENESS

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FIGURE 2. DETAIL VIEW OF ABRASION-MACHINE LAP SHOWING FEED SPOUT, CYLINDER IN WHICH PELLETS ARE HELD ON THE LAP, AND STANDARD PELLETS OF PRESSED TALC

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PRELIMINARY WORK TO DEVELOP A SUITABLE METHOD FOR MEASURING LUSTER

Preparation of a Set of Visually Rated Luster Standards

As a first step in the development of a method for measuring luster, a series of talc samples was prepared and submitted to individual observers who were asked to rate them according to their luster. In most cases, it was stated to the observers that luster did not necessarily imply whiteness or brightness; but was analogous to sheen, gloss, shininess, etc. No specific method for judging luster was suggested and no specific rating code was prescribed.

The results of this survey are shown in Table 1. The samples examined are listed in the left-hand column of the table and are described in footnotes. Each vertical column thereafter presents the ratings by the individual observers. The ratings are expressed numerically; the lower the number the higher the luster.

Some of the observers used a numerical system for grading the talc; most did not. For example, one employed such categories as "best", "next best", "poor", and "no good" (Observer E). For presentation in the table, these particular categories have been expressed as 1, 2, 3, and 4, respectively. Another observer (H) divided the samples into two groupings, "lustrous" and "inferior". These have been expressed as 1 and 2, respectively, in the table.

No observer attempted to distinguish between individual samples but reported his findings in groups. For example, Observer A listed Samples V, P, T, U, and L-7 as having the highest luster, with Samples M and Q in second place, Samples Y, S, and W in third, Sample W-2 in fourth, and Samples Z, X, and R in last. Observer B did select Sample V alone as being the most lustrous, but divided the remaining samples into groups.

The results indicate that:

- (1) Samples V, P, T, and U were almost unanimously judged to be the most lustrous.
- (2) Samples W, Q, S, M, and L-7 were judged by most observers to possess an intermediate luster.
- (3) Samples Y, Z, X, R, and W-2 were rated as having the least luster.

Physical Measurement of the Luster Standards

With this suite of samples, visually rated for luster, the research effort was next directed toward determining whether any of the available methods of optical measurement would yield results that coincided with those of visual observation. The methods tried are discussed in the following sections.

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an empirical quantity that has been found useful in determining the relative gloss of low-gloss papers and, reportedly, yields data which agree with those of visual observation. The results of the contrast-gloss measurement of the samples prepared in the manner described, were somewhat promising, in that all of the samples visually judged to possess the highest luster yielded contrast-gloss values in the highest range. There were, however, some anomalies in the results on the intermediate- and low-luster samples. Several of these judged visually to be almost chalky, exhibited high contrast-gloss values on the reflectometer. These were, notably, cyclone-overflow products, which were extremely fine. It was concluded that this method would not be satisfactory, not only because of the few anomalies but also because the sensitivity of the method was poor, i.e., the spread in readings between high-luster and low-luster material was quite small. The results are also shown in Table B-1 in Appendix B.

Contrast Gloss of Thin, Oriented Talc Surfaces

Contrast-gloss measurements were then made on samples rubbed onto filter paper, using the Gardner Multipurpose Reflectometer. The procedure of rubbing the talc onto filter paper was suggested by the practice employed by most observers in judging luster, which was to rub the talc onto their skin and to observe its reflectance at various angles. The results of these measurements agreed very closely with those of visual observation. Moreover, reasonably wide-spread readings existed between the readings for high-luster and for low-luster materials. The results are shown in Table B-1 in Appendix B.

PROCEDURE DEVELOPED FOR MEASURING THE CONTRAST GLOSS OF TALC SAMPLES

The procedure tentatively adopted for determining contrast gloss on talc samples is as follows:

Approximately 2 grams of talc are poured onto the center of a 12.5-centimeter Whatman No. 41-H filter paper. The talc is rubbed onto the paper with the middle finger or several fingers in a circular motion. The pressure applied in rubbing is approximately one-half pound as determined by preparing a sample on the pan of a laboratory beam balance. In rubbing the talc onto the paper, care should be taken to ensure that the finger is always moving excess talc about the surface. Experience has shown that unless excess talc is available, the rubbing action removes some talc that has already been aligned on and embedded in the surface and leads to lower contrast-gloss values. From 15 to 20 circular strokes are employed in rubbing.

The excess talc is poured from the paper back into the sample container, the paper is given a slight, sudden flick by a wrist movement and the surface is blown on gently to remove extremely fine talc. The flicking and blowing steps are necessary to ensure maximum reproducibility of results. If they are not used, contrast-gloss measurements may be low and erratic. The reason for this is not known for certain, but it is believed that rubbing alone without flicking or blowing results in a surface of properly oriented particles of talc but one in which fine unoriented particles lie on top of the oriented particles.

Specular Gloss

Specular gloss measurements were made on portable gloss meters of the kind commonly used to determine the gloss of painted surfaces. These measurements were made on bulk material contained in a shallow mold. The sample was lightly compacted, smoothed and leveled, by drawing a piece of plate glass across the surface. The specular gloss of the samples, thus prepared, was measured at three different angles of incidence and corresponding reflectance; 20°, 60°, and 85°. The method proved unsatisfactory because even those samples which had been judged visually to be the most lustrous gave readings of only 1 to 2 on the 100-unit scale.

Reflectance

Luminous apparent-reflectance measurements on bulk materials prepared as described above were made with the Gardner Multipurpose Reflectometer*. These measurements are approximately the same as the Rd measurements obtainable by the Gardner Color Difference meter which has been used in the previous work on talc.

- There was no correlation between the luminous apparent-reflectance data and the results of the visual luster-ratings of the samples. The data for the luminous apparent-reflectance measurements are shown in Table B-1, Appendix B.

Whiteness and Yellowness

Reflectance measurements were also made through tristimulus filters (i.e., reflectance with green, blue, and amber light) on samples prepared as above, using the Gardner Multipurpose Reflectometer**. From the data thus obtained, numerical expressions of whiteness and yellowness were calculated. Whiteness could not be correlated with the results of visual judgment. There was an apparent correlation in some instances with yellowness. Some of the more lustrous samples were, by the numerical convention, more yellow than less lustrous samples. However, the correlation was otherwise poor. It is believed that the yellowness of the more lustrous samples has been caused by some factor associated with the processing of the talc and is completely independent of the intrinsic optical characteristics of the crystals. Table B-1 in Appendix B shows the results of the measurements made with tristimulus filters and the derived whiteness and yellowness values.

Contrast Gloss of En Masse Samples

Contrast-gloss measurements on samples prepared as above (i.e., by dusting samples into a shallow mold and leveling and smoothing the surface by drawing a piece of plate glass across it) were made with the Gardner Multipurpose Reflectometer***. The contrast-gloss measurement on this machine is, in effect, specular reflectance divided by diffuse reflectance and is obtained by simultaneously measuring specular reflectance at +45, -45°, and diffuse reflectance at +45, 0°, with a green filter. It is

*See Appendix A, Item 1, for a description of the instrument and the procedure for determining reflectance. See also Item 2, page 611, for a discussion of the luminous apparent-reflectance measurement.

**Appendix A, Item 1, also Item 2, page 612.

***See Appendix A, Item 1, page 6, and Item 2, page 614 for discussion of the contrast-gloss measurement.

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As the procedure is applied in the Battelle laboratory the filter paper retains about 0.2 to 0.3 gram of talc. After the blowing step, the filter paper is placed on top of a stack of 10 or more 41-H papers which serve as a cushion and a light barrier. The 41-H paper is sufficiently translucent, so that if only a few are used somewhat erratic results will be obtained. Contrast gloss is determined as described on page 5, Item 1, in Appendix A.

Four measurements are made on each sample. After the first measurement, the paper is rotated approximately 90° and an additional reading is made. This is repeated twice more at 90° intervals. The four readings are averaged and the average is reported as the contrast-gloss value for the sample. Because no absolute standard is available, the Reflectometer is adjusted prior to running the samples so that when 41-H filter paper alone is used, the readings fall in the range between 1.40 and 1.44. Alternatively, the instrument may be adjusted so that the special ceramic plaque provided with the particular Battelle instrument will give a contrast-gloss reading of 1.82.*

Precision of the Contrast-Gloss Measurement

Table 2 presents data illustrating the precision of the method. Three groupings of data are shown in the table. The first two groups show the results obtained by a single operator who made five consecutive determinations according to the tentatively prescribed method on a talc sample with an intermediate contrast gloss and on one with a contrast gloss in the high range. As shown in the table, the individual readings exhibited a fairly wide spread. In the measurement of the sample with intermediate gloss, the readings obtained ranged from 1.46 to 1.51. It is Battelle's opinion, although it has not been firmly substantiated, that such divergence in the individual readings is legitimate and that it reflects actual differences in contrast gloss in the various areas of the sample being examined. It is entirely conceivable that the relatively crude technique of rubbing talc onto the filter paper and of eliminating excess talc by flicking and blowing does not result in a surface homogeneous with respect to contrast gloss. The average of the four individual readings probably is the fairest way to report the contrast gloss of a given sample. It has also been found that the average of four readings on a properly prepared sample does not deviate significantly from an average of 20 or more readings.

The agreement between the average results of each of the five determinations made on the sample with intermediate luster was reasonably good. The average deviation from the mean for these five samples was 0.011. The probable error calculated from this value is 0.007 and the reasonable limit for reporting determinations of contrast gloss on talc samples with intermediate gloss is calculated to be ± 0.02 .

On the five determinations shown in the second grouping in the table made on the talc sample with the higher contrast gloss, the spread in individual readings was considerably greater than on the talc sample with intermediate contrast gloss. Calculations based on these data indicate that the probable error of a single determination of contrast gloss of such samples is 0.011 and that it is reasonable to report results as ± 0.03 .

*After this report was in preparation, the Sponsor requested Battelle to investigate the possibility of obtaining primary standards for contrast gloss which could be used to standardize the measurement and permit interlaboratory check determinations on different instruments. It was not until the report was completely reproduced that satisfactory standards were obtained. See Appendix E for an account of the acquisition of the standards, the results of a limited number of contrast-gloss measurements made on the reflectometer after it had been adjusted to give readings consistent with the standards, and for the significance of the standardization of the measurement.

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(2) Working standards for contrast gloss should be calibrated on a goniophotometer*. Only with such standards can a close degree of accuracy be assured. Such a standard was not prepared for this study, recognizing that internally consistent data would suffice for the purpose of comparing one talc with another and further recognizing that the effects of any refinement to impart accuracy would be vitiated by the effect of the filter-paper backing.

Accordingly, the contrast-gloss data are not accurate, if one defines accuracy as agreement with objective truth. At best, and this is sufficient for the aims of the study, the method for determining contrast gloss may be likened to measuring with a yardstick on which the graduations begin at x and run $x + 1$, $x + 2$, etc.

Possibility of Refining the Method

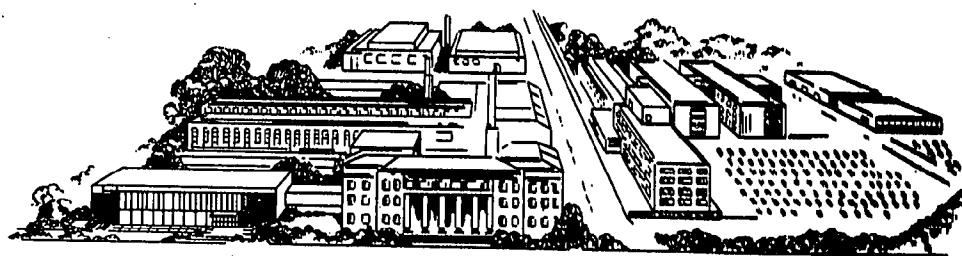
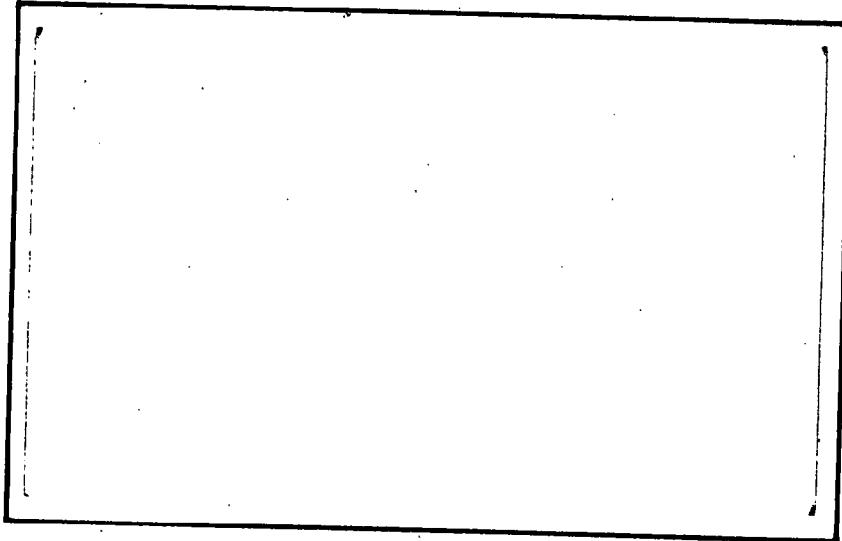
The most outstanding apparent weakness of the prescribed method is in the manner in which the talc surface is applied to the filter paper, i.e., by rubbing, flicking, and blowing. Two alternative methods were investigated. In the first of these, the talc was applied to the paper by means of so-called coating rods which are commonly used in ceramic laboratories to apply even coatings of ceramic slurries to surfaces. A coating rod consists of a stainless steel rod, tightly and closely wound with very fine stainless steel wire. In using it, one sets the rod horizontally at one edge of the surface to be coated, applies an excess of the coating material across the surface, and adjacent to the rod, and then applying slightly downward pressure evenly draws the rod through the material. This technique did not work for preparing talc surfaces. The readings for contrast gloss obtained on samples thus prepared were erratic and considerably lower than those obtained on the same samples prepared as prescribed in the method. It is probable that the coating rods which work well with ceramic slurries as a means for obtaining a uniform coating are unsatisfactory for use with dry materials and do not coat the surface completely. This would account for erratic results. It is also probable that the technique of drawing the wire-wound rod across a surface of talc fails to orient the talc particles as well as does the rubbing action with the fingers. This would account for the low contrast-gloss data obtained.

One method of applying the talc that showed some promise was one in which the talc was slurried in alcohol and then poured onto the paper, set in a vacuum filter funnel. The alcohol was filtered off and the paper dried by suction. In many cases, the readings for contrast gloss on samples prepared in this manner agreed closely with those prepared according to the prescribed method. In many other cases, results were erratic, probably because the action of pouring a slurry onto the paper did not necessarily result in an evenly spread properly oriented bed of talc, and also possibly because in handling the dried filter paper, the talc, which is apparently less firmly embedded by filtration than when applied by rubbing, became dislodged. With additional work, this filtration method might be developed into a more scientifically acceptable procedure for applying talc to filter paper than the rubbing method. Because the rubbing method was much faster and yielded apparently satisfactory results, such development work was not considered justifiable at this stage of the program.

*Appendix A, Item 2, page 614.

Exhibit 27

RESEARCH REPORT



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BATTELLE FIELDS OF RESEARCH

AERONAUTICAL ENGINEERING	INSTRUMENTATION
AGRICULTURAL CHEMICALS	HIGH ALLOYS AND RARE METALS
ALLOY DEVELOPMENT	LUBRICANT TECHNOLOGY
ANALYTICAL CHEMISTRY	MACHINICAL ENGINEERING
APPLIED MATHEMATICS	METAL FINISHING
BIOCHEMISTRY	METALLURGICAL PROCESSES
BIOPHYSICS	MINERALOGY AND MICROSCOPY
BUILDING AND ENGINEERING MATERIALS	MINERALS PROCESSING
CATALYSIS AND SURFACE CHEMISTRY	MICROBIOLOGY
CERAMICS	NONFERROUS METALLURGY
CHEMICAL ENGINEERING	NUCLEONICS
CHEMICAL PROCESSES	OPERATIONS RESEARCH
CORROSION TECHNOLOGY	ORGANIC CHEMISTRY
COMPUTER ENGINEERING	ORGANIC COATINGS
ECONOMICS	PETROCHEMICALS
ELECTRICAL ENGINEERING	PETROLEUM ENGINEERING
ELECTROCHEMICAL ENGINEERING	PHYSICAL CHEMISTRY
ELECTROCHEMISTRY	PHARMACEUTICAL CHEMISTRY
EXTRACTIVE METALLURGY	PRODUCTION ENGINEERING
ELECTRONICS	PULP AND PAPER TECHNOLOGY
FERROUS METALLURGY	RADIOISOTOPES AND RADIATION
FOUNDRY PRACTICE	RELIABILITY ENGINEERING
FOOD TECHNOLOGY	REACTOR TECHNOLOGY
FUELS AND COMBUSTION	REFRACTORIES
GRAPHIC ARTS TECHNOLOGY	RUBBER AND PLASTICS
GLASS TECHNOLOGY	SEMICONDUCTORS AND SOLID-STATE DEVICES
HIGH TEMPERATURE METALLURGY	SYSTEMS ENGINEERING
HUMAN ENGINEERING	TEXTILES AND FIBERS
IMMUNOLOGY AND CANCER STUDIES	THEORETICAL AND APPLIED MECHANICS
INDUSTRIAL PHYSICS	THERMODYNAMICS
INFORMATION PROCESSING	WELDING AND METALS-JOINING TECHNOLOGY
INORGANIC CHEMISTRY	WOOD AND FOREST PRODUCTS

PROGRESS REPORT

on

THE PHYSICAL CONCENTRATION OF
TALC ORES - FLOTATION OF ITALIAN
RUN-OF-MINE TALC

to

JOHNSON AND JOHNSON

December 31, 1959

by

Whitman E. Brown

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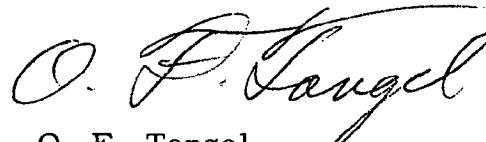
January 15, 1960

Mr. W. H. Ashton
Research Department
Johnson and Johnson
New Brunswick, New Jersey

Dear Mr. Ashton:

We are sending you six copies of our report on "The Physical Concentration of Talc-Ores - Flotation of Italian Run-of-Mine Talc", by Whitman E. Brown. This report, in conjunction with our similar report of July 31, 1959, on the "Flotation of Italian No. 2 Talc", gives the laboratory work that was the basis for the recommendation of a pilot talc-flotation plant and the data on which its design was based.

Sincerely yours,



O. F. Tangel

OF T:jvo

cc: Dr. W. H. Lycan
C. W. Swank

D E D I C A T E D T O T H E A D V A N C E M E N T O F S C I E N C E

TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION	1
SUMMARY - ITALIAN ROM TALC	3
SAMPLING AND MINERAL EVALUATION OF ITALIAN ROM TALC	4
EXPERIMENTAL WORK	4
Grinding.	4
Effect of Weight of Talc Charged to Pebble Mill in Wet and Dry Grinding.	6
Effect of Grinding-Media Weight.	8
Effect of Grinding Time	8
Effect of Pulp Density	8
Effect of Pebble Size.	12
Effect of Grinding Time on Production of Minus 10-Micron Particles	12
Simulated Closed-Circuit Grinding	15
Hydraulic Classification of Pebble Mill Product	17
Flotation	21
Flotation of Wet-Ground Minus 100-Mesh ROM Talc . .	21
Flotation of Dry-Ground Minus 200-Mesh ROM Talc . .	24
Flotation of Wet-Ground Minus 200-Mesh ROM Talc . .	24
Effect of HCl on Recovery and Quality.	24
Effect of Type of Frother on Recovery and Quality.	26
PROPOSED PILOT-PLANT FLOWSHEET	31
CONCLUSIONS	34
FUTURE WORK	36
APPENDIX	
SUMMARIZED RESULTS OF ALL FLOTATION TESTS MADE ON ITALIAN ROM TALC	A-1

B A T T E L L E M E M O R I A L I N S T I T U T E

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INTRODUCTION

This is the Third Progress Report on "The Physical Concentration of Talc Ores" and specifically applies to experimental results obtained from the beneficiation of Italian run-of-mine (ROM) talc. For comparative purposes, occasional references are made in the discussion about results obtained from Italian No. 2 talc.*

The objectives of the investigation were:

- (1) To obtain a product that consists essentially of talc platelets
- (2) To reject talc particles that are of a size and shape that create unpleasant dusting while talc is being dispensed from a container
- (3) To obtain a talc product with an obvious luster in order to convey to the consumer the immediate impression that the talc is of the highest quality
- (4) To investigate the variables that affect the grindability of talc

*Brown, W. E., "The Physical Concentration of Talc Ores - Flotation of Italian No. 2 Talc", Battelle Progress Report to Johnson and Johnson (July 31, 1959).

B A T T E L L E M E M O R I A L I N S T I T U T E

(5) To establish that the beneficiation process developed for Italian No. 2 talc is also applicable to the run-of-mine talc.

In addition to achieving the foregoing objectives, it was desirable that the finished talc product meet the following specifications:

Moisture: Not more than 0.15 per cent

Solubility in HCl: Not more than 6 per cent

Fineness: Not less than 99.7 per cent through a 100-mesh Tyler sieve and not less than 98.5 per cent through a 200-mesh sieve

Microscopic Structure: Platelets, and no acicular or excessive granular crystals

Bulk Density: Not less than 22 nor more than 27 pounds per cubic foot, when tested by the Scott Volumeter.

In further keeping with the standards of production, it is desirable that the finished talc product have essentially the same whiteness as that currently being marketed by Johnson and Johnson.

The methods of beneficiation employed in previous work were hydraulic cycloning and flotation. It was established in the earlier investigations that hydraulic cycloning alone not only rejected objectionable dust-forming particles, but also improved the platy content of the talc. However, cycloning alone was not sufficient to obtain the ultimate in purity. Flotation proved to be effective for the removal of more nonplaty talc, tremolite, dolomite, and other accessory gritty and off-color minerals. The combined cyclone-flotation processes developed for Italian No. 2 talc were not effective in improving the luster, as judged by casual observations, although the mineral particles were 98 to 99 per cent talc platelets. If a product consisting of such high-purity mineral particles did not have an improved luster, then a method of comminution, other than roller milling, might affect favorably the morphology of the platelets and the luster. Wet and dry pebble milling seemed logical methods to try.

In addition to obtaining a talc with an improved luster, certain economic factors had to be considered. It was the Sponsor's desire to remove from talcum powder objectionable dust-forming particles: platelets or other forms. Some experiments indicated that particles finer than 10 or 15 microns were easily airborne and, therefore, their removal should improve the powder. Size-distribution experiments, however, showed that about 27 per cent of the Italian No. 2 type of talc was finer than 10 microns and about 40 per cent of the weight was finer than about 14 microns, so that complete rejection of the potential dust, in the final analysis, would

increase the total cost of the raw material in direct proportion to the amount of rejected weight. This does not include the operating cost of the classification process. Battelle believed that carefully controlled wet-pebble-mill grinding might result in a ground product having less of the objectionable fine sizes. Johnson and Johnson was asked to obtain Italian run-of-mine (unground) ore samples, so that the problems related to luster and fine grinding might be investigated in the laboratory.

SUMMARY - ITALIAN ROM TALC

Batch wet grinding in a laboratory pebble mill produced fine-ground talc that had a higher order of luster than that obtained by dry roller milling or dry pebble milling.

Batch wet pebble milling resulted in faster grinding of minus 10-mesh talc through 200 mesh but produced more objectionable fine talc in the minus 10-micron sizes than batch dry pebble milling.

Variables that affect rates of grinding were investigated. It was found that a grinding time of less than 11 minutes and a circulating load of more than 336 per cent may be required to avoid overgrinding. Laboratory experiments were not successful in obtaining a ground product containing less than 35 per cent of the weight finer than 10 microns. Data were obtained, however, that can be used to determine the approximate grinding time necessary to obtain the desired results. This information is discussed with Figure 5 in the "Grinding" section of this report.

It is necessary to grind finer than 100 mesh, because a 100-mesh grind yields products that are gritty, although the high luster is remarkably evident.

If the ROM talc is overground, that is, contains more than about 40 per cent of minus 10-micron particles, one stage of cyclone classification may not be sufficient for satisfactory removal of the fines. However, ROM talc which has not been overground responds satisfactorily to cyclone classification.

Flotation of wet-ground and classified ROM talc was successful. Beneficiated products were obtained that were 97 to 99 per cent platy talc. The yield expected in a continuous operation is 80 per cent of the weight of the flotation feed; this amounts to 56 per cent of the original ore.

The beneficiated talc was an improvement in all respects when compared with the Johnson and Johnson specifications for the raw material currently used in their marketed Baby Powder.

The beneficiation process developed for Italian No. 2 talc is also satisfactory for Italian ROM talc when the proper crushing and grinding equipment are used.

SAMPLING AND MINERAL EVALUATION OF
ITALIAN ROM TALC

Johnson and Johnson arranged for a sample of coarse run-of-mine (ROM) Italian talc to be shipped to Battelle for grinding and flotation experiments, including luster studies of the products from the experiments. Approximately 4,450 pounds of ROM ore were received for this work in July, 1958. One bag containing 150 pounds of talc was reserved for miscellaneous purposes. The remaining portion, containing some pieces up to 1-1/2 inches maximum dimension, was mixed by coning and reduced in quantity by riffling to 268 pounds. The 268-pound sample was roll crushed through a 10-mesh Tyler screen for feed material in the grinding (pebble-milling) experiments. A small but representative portion of the minus 10-mesh talc was ground in a pebble mill to pass through a 200-mesh Tyler screen and examined with a microscope.

Results of the microscope analysis showed that the Italian ROM talc was almost identical to the Italian No. 2 talc in mineral composition. It contained about 90 per cent platy talc, 6 per cent nonplaty talc, 2 per cent dolomite, and 2 per cent tremolite. The Italian No. 2 talc contained about 90 per cent platy talc, 6 per cent nonplaty talc, 3 per cent dolomite, and 1 per cent tremolite.

Because of the nearly identical mineral compositions of the ROM talc and the Italian No. 2 talc, it was implied that beneficiation methods developed for Italian No. 2 material would probably be just as effective for the processing of ROM talc.

EXPERIMENTAL WORK

Grinding

The objectives for grinding ROM talc were threefold:

- (1) Grind the ore through 200 mesh in a manner that would result in the production of less minus 10-micron talc than that contained in Italian No. 2 talc, or significantly less than about 27 per cent

(2) Grind the ore in a manner that would result in a ground product having a more prominent luster than that obtainable from Italian No. 2 talc

(3) Determine the most expedient method of grinding to produce ground products suitable for subsequent classification and flotation experiments in the laboratory.

Because talc is one of the softest natural minerals, one might expect that it would grind easily and overgrinding would be difficult to avoid.

Preliminary experiments on wet grinding minus 10-mesh talc through 200 mesh indicated that grinding of talc was not so simple as grinding the granular type of minerals.

The reasons are that (1) talc resists grinding because of its lubricity and (2) the high specific surface of liberated talc platelets per unit of weight fixes the maximum grinding density of the slurry at about 45 per cent solids. If the slurry density is increased a few per cent beyond this point, it becomes a sticky paste, which is an impracticable condition for grinding.

Because of the unusual grinding characteristics of talc, specifically the Italian type, it became necessary to determine what conditions would be required to grind efficiently and what variables have the most influence on the desired results.

A program for grinding experiments was established to investigate the influence of certain variables on the efficiency in producing minus 200-mesh talc. The variables investigated were:

- (1) Effect of weight of talc charged to pebble mill in wet and dry grinding
- (2) Effect of grinding-media weight
- (3) Effect of grinding time
- (4) Effect of pulp density
- (5) Effect of pebble size.

The pebble mill used for these experiments is made of porcelain with inside dimensions of 7-1/2-inch diameter and 7-1/4 inches long. It was rotated at 70 rpm.

Effect of Weight of Talc Charged to Pebble Mill in
Wet and Dry Grinding

Figure 1 shows the effect of the weight of talc charged to the pebble mill on the per cent of talc reduced to minus 200 mesh.

There are two prominent characteristics evident from the data plotted in Figure 1.

- (1) Wet grinding is more effective than dry grinding in reducing the particle size of talc to finer than 200 mesh.
- (2) The percentage of talc reduced to 200 mesh and finer decreases at a constant rate with an increase in the amount of talc charged to the pebble mill.

The data show that, with a charge of 120 grams of talc, which is about 24 per cent of the nominal mill capacity, about 91 per cent of the charge was reduced to 200 mesh in a grinding time of 60 minutes. If, however, the charge is increased to 240 grams, or about 48 per cent of capacity, the amount of talc that is reduced to 200 mesh is about 73 per cent. Finally, if the talc charge is increased to 500 grams, a 60-minute grind will reduce 35 per cent of the charge to 200 mesh.

Dry-grinding characteristics are similar to wet-grinding characteristics with respect to rate of change in grinding with increased loading. However, when a 120-gram charge of talc was wet ground for 60 minutes, about 91 per cent of the slurry was finer than 200 mesh. After dry grinding the same weight of charge, only about 71 per cent of the resulting powder was finer than 200 mesh.

The data plotted in Figure 1 do not immediately reveal all of the facts. Actually, the amount of minus 200-mesh material produced reached a peak when a charge of 400 grams was wet ground or dry ground. The following tabulation of grinding data illustrates this point.

Weight of -10 Mesh Talc Charge, grams	Per Cent of Charge Ground Finer Than 200 Mesh				Total Weight of Charge Ground Finer Than 200 Mesh, grams	
			Wet	Dry	Wet	Dry
	Wet	Dry	Wet	Dry	Wet	Dry
120	120	90.6	70.5	108.7	84.6	
240	240	73.0	59.0	175.2	141.6	
400	400	52.7	42.0	210.8	168.0	
500	500	39.0	32.0	195.0	160.0	

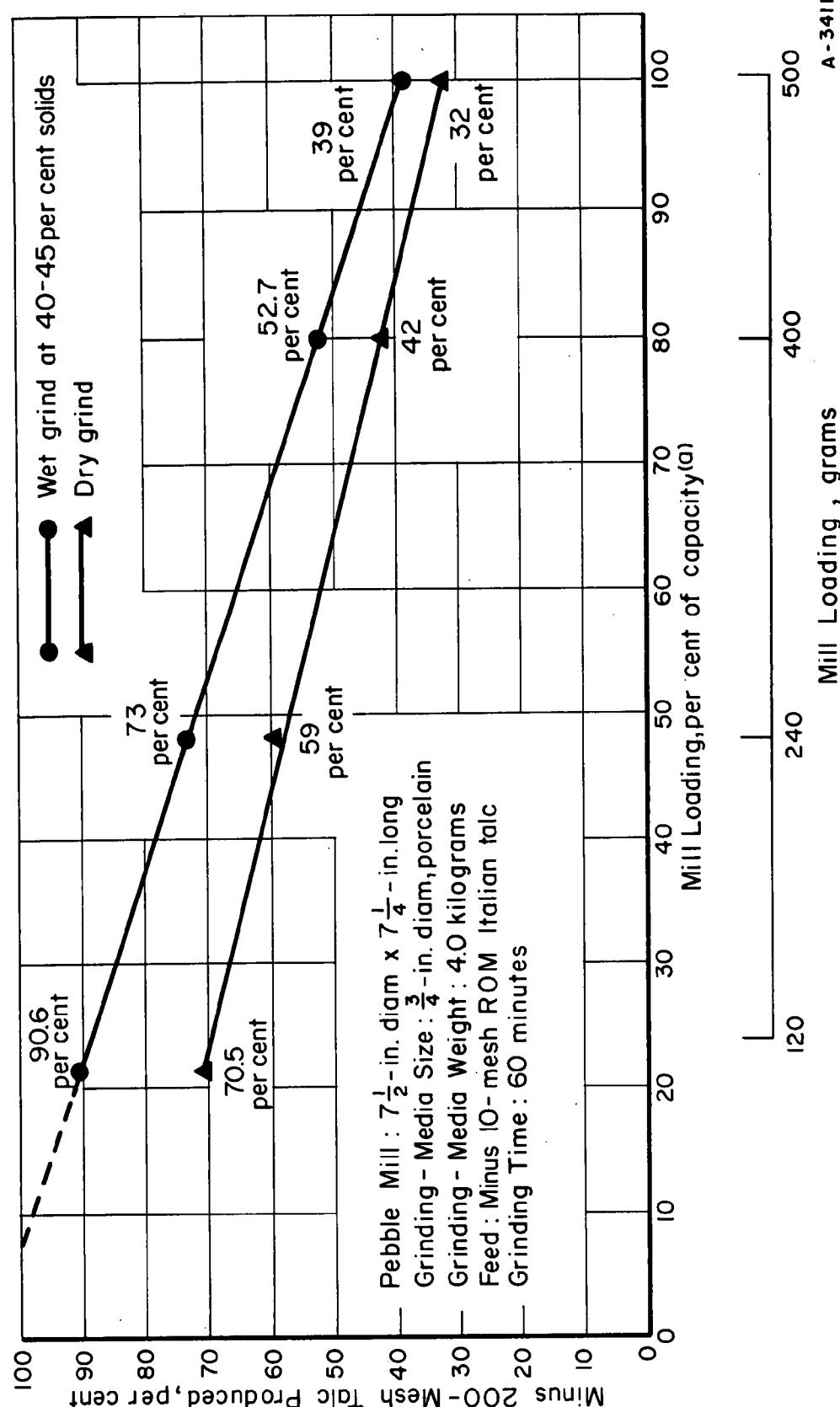


FIGURE 1. THE EFFECT OF THE WEIGHT OF TALC CHARGED TO THE PEBBLE MILL ON THE PER CENT OF TALC REDUCED TO MINUS 200 MESH

(a) Pebble-mill capacity considered to be 500 grams.

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Effect of Grinding-Media Weight

Figure 2 shows the effect of the weight or volume of grinding media on the per cent of talc reduced to minus 200 mesh. The curve shows that, as the weight of grinding media was increased from 2.5 kilograms, or about 31 per cent of the mill volume, to 5.0 kilograms, or about 62.5 per cent of the mill volume, the amount of talc that was reduced to 200 mesh was increased from 70 per cent to about 95 per cent. The increased rate of grinding is pronounced as the amount of grinding media is increased up to 50 per cent of the mill volume. Increasing the grinding media in excess of 50 per cent of the mill volume resulted in small increases in producing additional amounts of minus 200-mesh talc. Furthermore, it is not possible to have more than 50 per cent of the mill volume occupied by grinding media in a continuous operation.

Effect of Grinding Time

The next series of experiments were made to determine the effect of grinding time on the amount of talc reduced to 200 mesh. These experiments were made by wet and dry grinding 120-gram charges of minus 10-mesh talc over a range of 15 to 90 minutes.

The data obtained from these experiments are plotted in Figure 3 and show that, as the wet-grinding time is increased from 15 to 60 minutes, the amount of talc reduced to 200 mesh is increased from 50 to 90 per cent. Grinding in excess of 60 minutes resulted in only minor increases in the amount of minus 200-mesh talc produced and appears impractical. Figure 3 also shows that wet grinding is more effective than dry grinding. Although the wet- and dry-grinding curves tend to parallel each other, wet grinding produced from 15 to 30 per cent more 200-mesh material for any given grinding period.

Effect of Pulp Density

Previous experiments established that, when a large percentage of the talc is ground finer than 200 mesh, pulp densities near or in excess of 45 per cent solids resulted in a pasty, nonfluid mass. Experiments were made on grinding 120 grams of minus 10-mesh talc charges at 30, 35, 40, and 45 per cent solids. The resulting data are plotted in Figure 4 and show a gradual increase in grinding efficiency as the per cent solids of the slurry is increased from 30 to 45 per cent. At 30 per cent solids, about 72 per cent of the talc was ground through 200 mesh, and at 45 per cent solids the ground product was about 91 per cent minus 200 mesh.

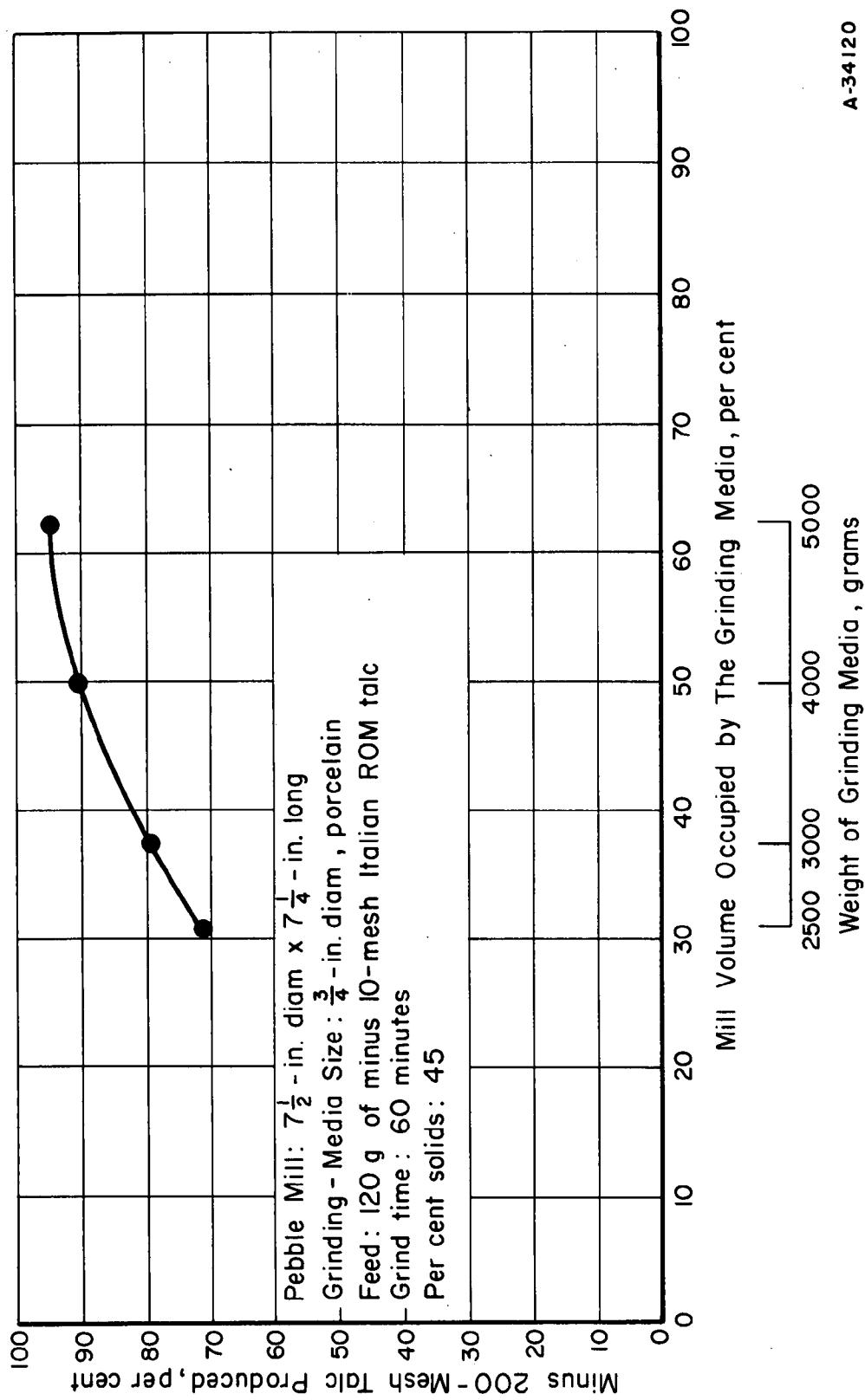


FIGURE 2. THE EFFECT OF THE WEIGHT OR VOLUME OF GRINDING MEDIA ON THE PER CENT OF TALC REDUCED TO MINUS 200 MESH

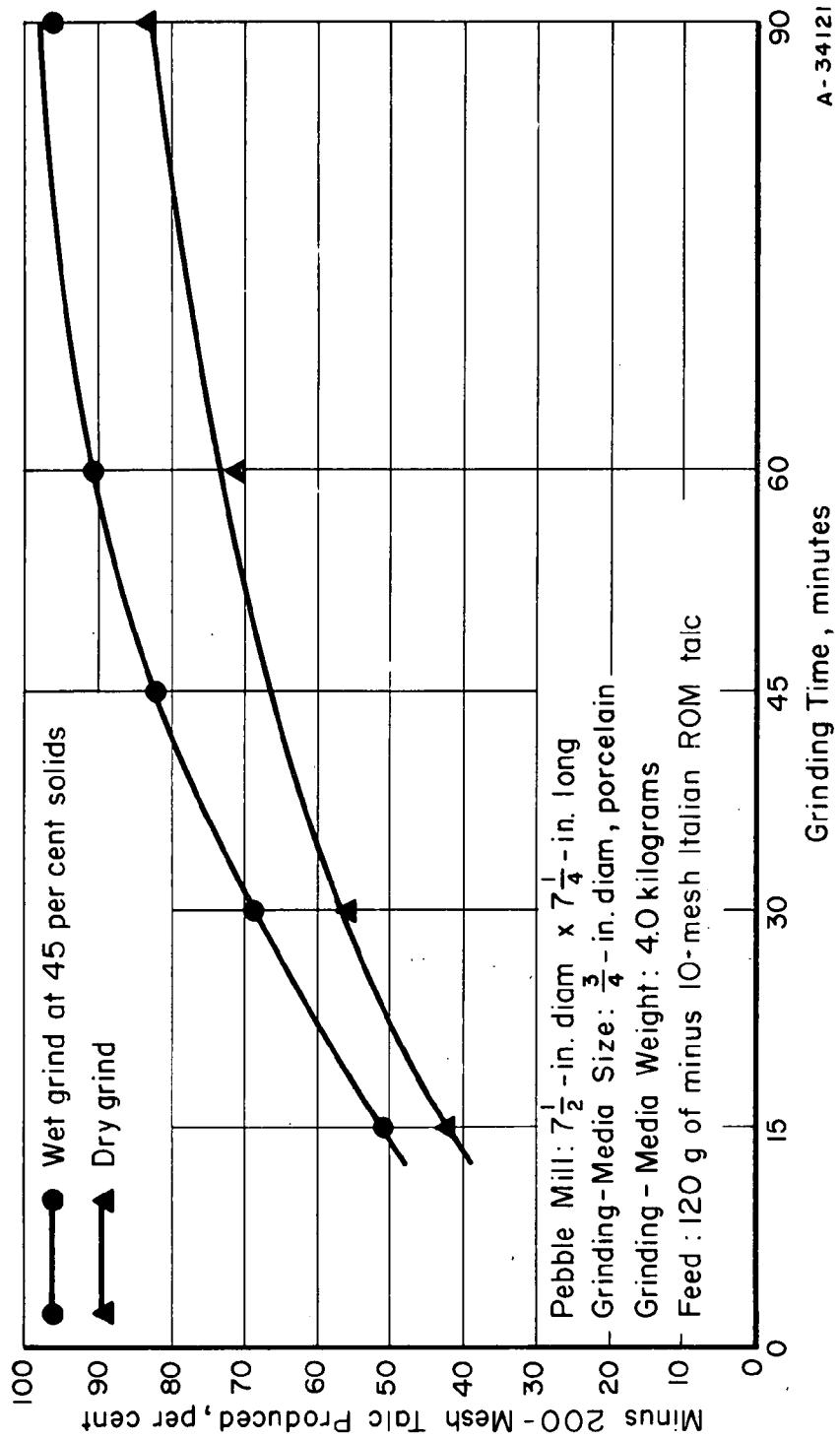


FIGURE 3. THE EFFECT OF GRINDING TIME ON THE PER CENT OF
TALC REDUCED TO MINUS 200 MESH

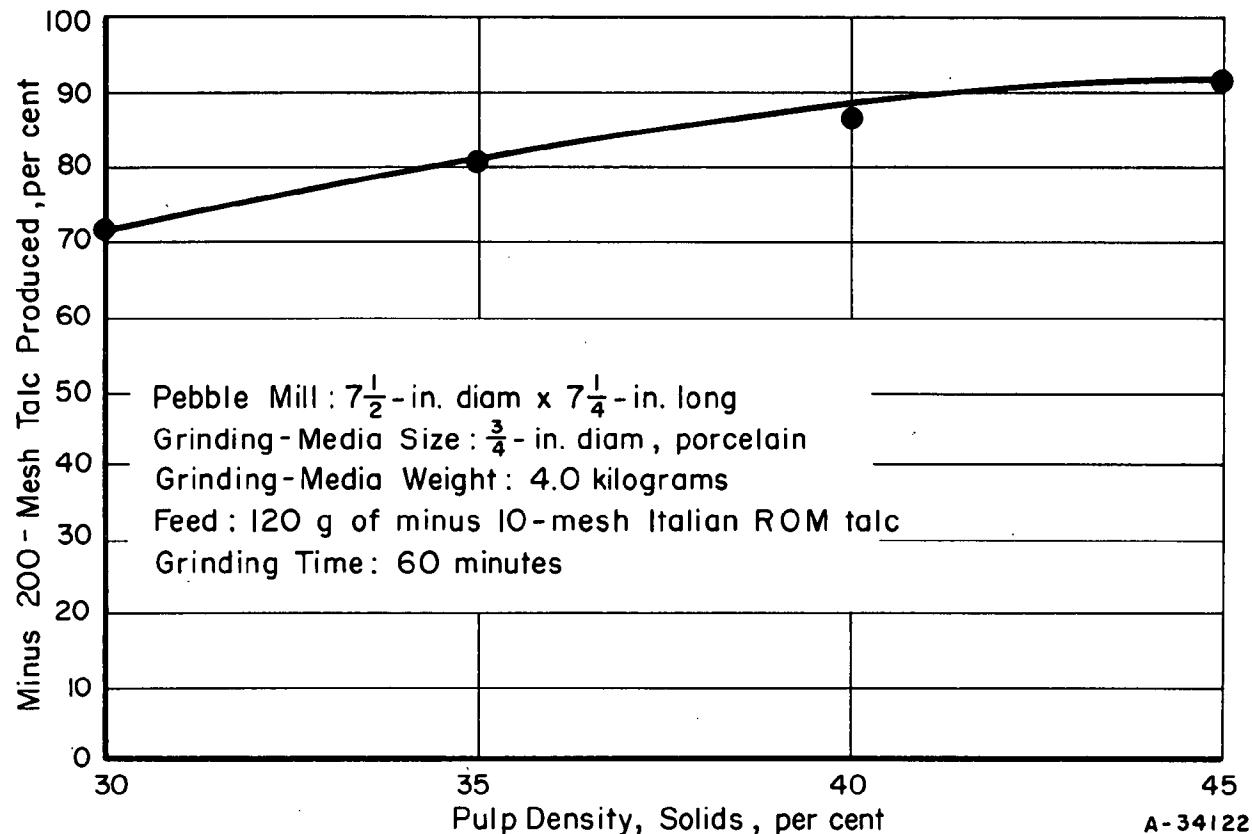


FIGURE 4. THE EFFECT OF THE PULP DENSITY DURING
GRINDING ON THE PER CENT OF TALC
REDUCED TO MINUS 200 MESH

Effect of Pebble Size

This part of the investigation was limited to pebble sizes of 0.75-inch diameter, 1.5-inch diameter, and a mixture of the two. Experiments were made using 120 grams of minus 10-mesh talc charges adjusted with water to 45 per cent solids and ground for 60 minutes.

The following tabulation gives the data obtained from this work:

Size of Pebbles, inches	Weight of Pebbles, kilograms	Minus 200-Mesh Talc Produced, weight per cent
0.75	4.0	90.6
0.75 and 1.50(a)	4.0	88.5
1.50	4.0	86.8

(a) About 50 per cent of each size.

These data show that small-diameter pebbles will grind more talc through 200 mesh than large-diameter pebbles in a given length of time. Four kilograms of 0.75-inch pebbles reduced 90.6 per cent of the 120-gram talc charge through 200 mesh. The same weight of 1.5-inch pebbles reduced 86.8 per cent of the charge through 200 mesh.

Effect of Grinding Time on Production of Minus 10-Micron Particles

In the foregoing discussion about the various factors involved in grinding, no effort was made to determine the amount of minus 10-micron talc produced. This was partly because optimum grinding conditions were not known and also because it was planned to combine the most effective grinding conditions and run a series of experiments, using time as the only variable, and obtain more complete size-distribution data. The following grinding conditions were selected as desirable:

Weight of Pebble Charge	4.0 kilograms
Size of Pebbles	0.75 inch
Weight of Talc to Be Ground	120 grams
Solids Content of Slurry	40 per cent

The essentials of the experimental procedure consisted of grinding a charge for 15, 30, 45, and 60 minutes. At the end of each time period, the entire charge was removed from the pebble mill and washed off the pebbles. The ground slurry was screened on a 200-mesh Tyler sieve and the minus 200-mesh portion was treated by sedimentation to remove the minus 10-micron particles. (Minus 10-micron sedimentation time was based on the rate of settling of 10-micron quartz particles.) The three sized products, plus 200 mesh, minus 200 mesh plus 10 microns, and minus 10 microns, were dried and weighed. The resulting data are plotted in

Figure 5. The three curves shown in the figure permit determination of the amount of any of the three size ranges present in the ground talc at any given time.

It is interesting to note that, after a 60-minute grind, only about 11 per cent of the talc was coarser than 200 mesh, 42 per cent was in the desired size range of minus 200 mesh plus 10 microns, and about 44 per cent was finer than 10 microns. One of the grinding objectives was to produce less than about 27 per cent of minus 10-micron particles and, since a 60-minute grind produced 44 per cent, the time of grinding was much too long. If some shorter time of grind is selected, say 15 minutes or 11 minutes, the desired results are almost obtained. Table 1 shows the weight distributions of the ground products at the end of 15 minutes and of 11 minutes.

TABLE 1. DISTRIBUTION OF SIZES AFTER 15- AND 11-MINUTE WET-GRINDING PERIODS OF ROM ITALIAN TALC

Sized Product	Distribution			
	Weight Per Cent of Total Product		Weight Per Cent After Removal of Plus 200-Mesh Fraction	
	15 Min	11 Min	15 Min	11 Min
+ 200 mesh	48	58	0.0	0.0
- 200 mesh + 10 micron	36	30	69.2	71.5
- 10 micron	16	12	30.8	28.5
Total	100	100	100.0	100.0

The data given in Table 1 show that 11 minutes should give about the same grind as that found in Italian No. 2 talc. That is to say, after removal of the plus 200 mesh (which in normal operation would be returned to the pebble mill for further grinding), the minus 200-mesh talc contained 28.5 per cent of minus 10-micron particles, compared with about 27 per cent in Italian No. 1. A still shorter time of grind appears necessary, and calculations for a 6-minute grind show that the minus 200-mesh talc would contain about 25 per cent of minus 10-micron particles.

As the time of grind is decreased, the amount of plus 200-mesh talc to be reground is increased and, in a continuous operation, would increase the circulating load. It is common commercial practice to use large circulating loads to minimize overgrinding.

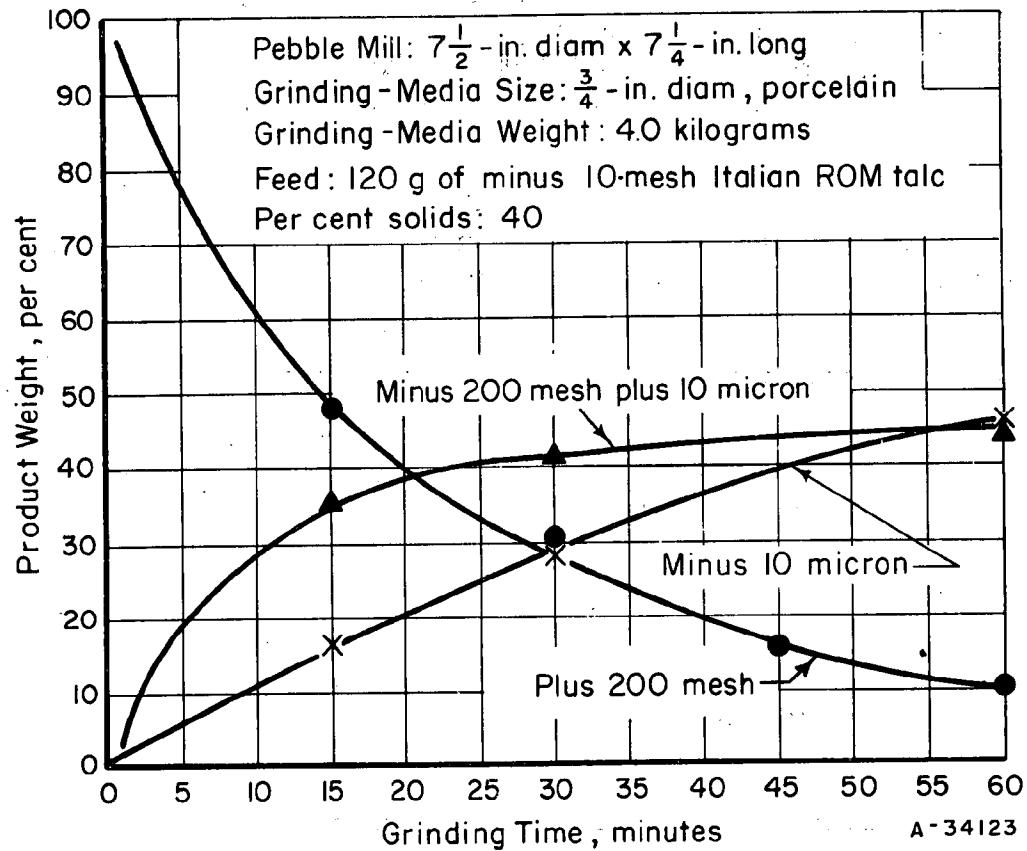


FIGURE 5. THE EFFECT OF GRINDING TIME ON THE PER CENT OF MINUS 200-MESH PLUS 10-MICRON AND MINUS 10-MICRON TALC PRODUCED

The data given in Table 1 and Figure 5 must be used as a guide only, but should be useful for making reasonable estimates for grinding performance and trends.

Grinding studies were carried a step further at a later date (after a number of flotation tests had been made) but are discussed here for conformity.

Simulated Closed-Circuit Grinding

Investigations were made on wet grinding that simulated continuous closed-circuit grinding, although actually consisting of batch grinds of short duration. A circulating load consisting of the unground plus 200 mesh was returned to the mill for further grinding, as is common in continuous-grinding practice.

The essential parts of the procedure were to add a given weight of charge to the pebble mill, grind for a specified time, and screen the ground product on a 200-mesh sieve. The plus 200-mesh portion was returned to the mill as a circulating load and a new amount of minus 10-mesh feed, equivalent in weight to the minus 200 mesh produced, was added to the pebble mill. This was repeated several times, until the mill charge came to equilibrium or, in other words, until the amount of minus 200 mesh produced from each grind was about the same weight as in the preceding grind. The foregoing procedure was followed for grinding periods of 15 minutes on a 120-gram talc charge and 11 minutes on a 240-gram charge. The ground products were separated at 200 mesh and 10 microns in the usual manner, by screening and sedimentation. The results obtained are given in Table 2.

Table 2 shows two separate sets of grinding conditions and the resulting distribution of sizes in the ground products. After a 120-gram charge was ground for 15 minutes, the resulting slurry contained 18 per cent of the weight in particles finer than 10 microns. Only 29.9 per cent of the talc was in the desired particle-size range of minus 200 mesh plus 10 microns. About 52 per cent of the ground product was returned to the pebble mill as a circulating load. After the plus 200-mesh portion was screened out, the minus 200-mesh slurry contained 37.5 per cent of the weight finer than 10 microns. This was considered as overgrinding, because one of the objectives was to grind in a manner that would result in less than 27 per cent of the weight finer than 10 microns.

In order to overcome the excessive overgrinding, an experiment was made that would increase the circulating load substantially. The amount of talc initially charged to the mill was increased to 240 grams and the grinding time was shortened to 11 minutes. The size distribution of the ground product for the 11-minute grinding period shows that 8.0 per cent of

TABLE 2. RESULTS OF A SIMULATED CLOSED-CIRCUIT
GRIND ON MINUS 10-MESH ROM ITALIAN TALC

Grinding Time, minutes	15	11
Mill, 7-1/2-in. diam x 7-1/4-in. long	Pebble	Pebble
Mill Speed, rpm	70	70
Weight of Pebbles, kilograms	4	4
Average Pebble Size, inches	3/4	3/4
Initial Talc Charged to Mill, grams	120	240
Minus 10-Mesh New Feed to Mill, grams	57.5	55
Plus 200 Mesh Returned to Mill, grams	62.5	185
Per Cent Solids in Mill	40	40
Circulating Load, per cent of new feed	108.7	336.3
Size Distribution of Pebble-Mill Discharge, per cent		
+ 200 Mesh	52.1	77.1
-200 Mesh + 10 Micron	29.9	14.9
- 10 Micron	18.0	8.0
Total	100.0	100.0
Size Distribution of Pebble-Mill Discharge After Removal of Plus 200-Mesh Minerals, per cent		
-200 Mesh + 10 Micron	62.5	65.0
- 10 Micron	37.5	35.0
Total	100.0	100.0

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the weight was finer than 10 microns. At first glance, it might appear that the objective had been accomplished. However, after removal of the plus 200-mesh portion of the ground product, the minus 200-mesh material contained 35 per cent of the weight finer than 10 microns. So it is noted that, although the circulating load was increased from 108.7 per cent for the 15-minute grind to 336.3 per cent for the 11-minute grind, and with a doubled charge, the net result is a reduction of minus 10-micron talc from 37.5 per cent to 35 per cent. The reason for such a slight change in the amount of 10-micron material produced is not fully understood. In order to arrive at the objective of producing less than 27 per cent of minus 10-micron talc in the minus 200-mesh product, more variables would have to be investigated, such as shorter grinding time, larger or mixed diameter pebbles, coarser size talc as pebble-mill feed, and perhaps increased dilution.

It was felt that grinding characteristics on a continuous basis, with coarser ore feed, larger diameter pebbles, and continuous classification, would all favor reaching the size-distribution objective; therefore, the laboratory grinding program was terminated.

Another objective of pebble milling was to obtain a product with a noticeably higher luster than that exhibited by the Italian No. 2 talc, which had been dry ground in a Raymond-type roller mill. The first wet-grinding experiment in the pebble mill was successful in this respect. In fact, all wet-grinding experiments gave ground products having a relatively high luster.

The next step in the investigation involved the separation of the minus 10-micron talc from the minus 200-mesh ground product.

Hydraulic Classification of Pebble-Mill Product

Particle-size classification of the pebble-mill product for removal of minus 10-micron particles was necessary in order to prepare a satisfactory flotation feed and at the same time to remove potential dust-forming minerals.

The ground ore from the pebble mill was screened on a 200-mesh Tyler sieve for removal of objectionable oversize talc. The minus 200-mesh portion was adjusted with water to 5 per cent solids by weight and cycloned in a 30-mm-diameter glass cyclone.

A complete description of the cyclone process and of various experiments on the classification of Italian No. 2 talc has been reported* to Johnson and Johnson. In that report, a cycloning procedure was described

*Brown, W. E., "The Physical Concentration of Talc Ores - Flotation of Italian No. 2. Talc", Battelle Progress Report to Johnson and Johnson (July 31, 1959).

and was considered an acceptable method for removal of objectionable fine particles. There was no reason to believe that a change in classification procedure was necessary. If the classification of the ROM ground talc were satisfactory, using the same procedure, it would aid in establishing that the classification part of the beneficiation process was applicable to both the Italian No. 2 and ROM talcs.

The pebble-mill wet-ground products, as reported in Table 3, were screened on 200 mesh and the minus 200-mesh portions were hydraulically cycloned for elimination of as much of the minus 10-micron particles as was practicable in one stage of classification.

In discussing the removal of minus 10-micron particles, it is necessary to qualify the purpose and the results. A preponderance of minus 10-micron particles in a flotation feed creates a voluminous froth that not only is difficult to break down, but also traps the undesirable minerals. Removal of minus 10-micron particles is further desirable because of their dusting potential. However, it is not necessary to remove all of the minus 10-micron particles to obtain a satisfactory froth, nor is it necessary to remove all of such particles to eliminate excessive dusting. Perhaps total dusting tendency never can be eliminated, because particles larger than 10 microns will be airborne occasionally. A compromise was accepted when a product was obtained that would contain about 10 to 12 per cent of minus 10-micron particles. Finally, it would be economically impractical to achieve absolute 10-micron-particle rejection, and it may even be mechanically impossible.

Therefore, in discussions of this nature, "removal of minus 10-micron particles", the meaning is that the product is treated in a manner that results in something less than about 10 to 12 per cent of the weight finer than 10 microns.

The cyclone classification of the ground products was tried with a feed pressure of 14.7 psi applied to the 15-minute-ground product and 14.7 and 23.0 psi applied to the 11-minute-ground product. The results of classification of these experiments are given in Table 3.

Table 3 shows that, when a 15-minute pebble-milled product was cycloned at 14.7-psi inlet pressure, an excessive amount, 48.5 per cent, of the feed weight was rejected in the cyclone overflow. Sedimentation analysis showed that 42 per cent of the weight of the overflow product was of particles larger than 10 microns. Therefore, 32.6 per cent of all the plus 10-micron talc in the cyclone feed was lost or rejected in the cyclone overflow.

When the product of an 11-minute grind was cycloned at 14.7-psi inlet pressure, the amount of cyclone overflow dropped to 36.7 per cent and contained 31.9 per cent by weight of plus 10-micron material.

TABLE 3. DISTRIBUTION OF WEIGHT AND PARTICLE SIZE OF HYDRAULICALLY CLASSIFIED
(CYCLOINED) MINUS 200-MESH PEBBLE-MILLED PRODUCTS

Operating Conditions:

Cyclone Diameter, mm	30
Feed Inlet Diameter, mm	6
Overflow Vortex Diameter, mm	11
Underflow Apex Diameter, mm	5.5
Feed Solids Content, per cent	5
Feed Volume Rate, gpm	
At 14.7 psi	2.7
At 23.0 psi	3.3

Grinding Time, min	Cyclone Product	Weight Per Cent	Weight Per Cent in Product		Distribution		Remarks
			-10 μ	+10 μ	-10 μ	+10 μ	
15	Feed	100.0	37.5	62.5	100.0	100.0	Feed pressure 14.7 psi
	Overflow	48.5	58.0	42.0	75.0	32.6	
	Underflow	51.5	18.2	81.8	25.0	67.4	
	Total	100.0	37.5	62.5	100.0	100.0	
11	Feed	100.0	35.0	65.0	100.0	100.0	Feed pressure 14.7 psi
	Overflow	36.7	68.1	31.9	71.4	18.0	
	Underflow	63.3	15.8	84.2	28.6	82.0	
	Total	100.0	35.0	65.0	100.0	100.0	
11	Feed	100.0	36.2	63.8	100.0	100.0	Feed pressure 23.0 psi
	Overflow	36.7	75.1	24.9	76.2	14.3	
	Underflow	63.3	13.6	86.4	23.8	85.7	
	Total	100.0	36.2	63.8	100.0	100.0	

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Therefore, the amount of plus 10-micron particles lost to the overflow is reduced from 32.6 per cent in the 15-minute grind to 18 per cent in the 11-minute grind.

Further reduction in loss of plus 10-micron particles was achieved by increasing the cyclone feed pressure to 23 psi; at this pressure, 14.3 per cent by weight of the plus 10-micron particles was lost.

Although the foregoing discussion pertains to recovery or distribution of the plus 10-micron particles, it is equally important to know how effective was the rejection of the potential dust, or minus 10-micron particles. Table 3 shows that the highest rejection of minus 10-micron particles to the cyclone overflow was obtained from the cycloning of an 11-minute pebble-mill-ground product at 23-psi cyclone feed pressure. The cyclone overflow contained 76.2 per cent of all the minus 10-micron particles contained in the cyclone feed. Although 23.8 per cent of all the minus 10-micron particles were in the cyclone underflow, this was offset by the high recovery of the plus 10-micron particles. As a result, the cyclone underflow representing 63.3 per cent of the feed weight contained only 13.6 per cent of minus 10-micron particles.

The summary of this work is:

- (1) Classification for 10-micron-particle separation was more effective on the shorter grind, that is, 11 minutes.
- (2) Classification for 10-micron-particle separation was more efficient when the feed pressure was increased from 14.7 to 23.0 psi. This is true for both the best recovery of plus 10-micron particles and highest rejection of the minus 10-micron particles.

Although the data indicate that cyclone feed pressures in excess of 23 psi might result in still higher efficiencies of separation, it is probable that with higher pressures the weight per cent of the overflow product may increase.

Another point worthy of mention is that 14.3 per cent of the original plus 10-micron particles are lost to the cyclone overflow. If this cyclone overflow were to be treated in a second stage of cyclones, some of the plus 10-micron fraction would be recoverable. The amount recoverable, without including an undesirable amount of minus 10-micron-particle weight, probably would not exceed 85.7 per cent of 14.3 per cent, or 12.3 per cent. It certainly should not be less than 6 per cent. Assuming that 60 per cent of the flotation feed weight is recovered, the potential over-all increase is from about 4 to 7 per cent of the original weight of the talc.

Flotation

Microscope examination of the ROM head sample revealed that the ore was mineralogically the same as Italian No. 2 talc. Because of the similarity of the two samples, it was believed that the beneficiation procedures of classification at about 10 microns followed by flotation of the plus 10-micron product (cyclone underflow) would be effective when the same flotation conditions were applied.

Generally, the flotation conditions that had given good results with Italian No. 2 talc were pulp densities in the range of 5 to 10 per cent solids, and hydrochloric acid and Dowfroth 200 or 250 as reagents. Hydrochloric acid was used both to neutralize the slurry and as an aid in the depression of fine-size particles. Dowfroth 200 or 250 was selected as the talc frother-collector because it is totally water soluble, requires a minimum of conditioning time, and has no collecting properties for other than the natural-floating-type minerals. Some of the advantages of the Dowfroths are that they do not leave any residual odor or discoloration on dried mineral products, nor do they chemically attack metal or rubber to any significant degree.

Experiments were made on the talc wet-ground through 100 mesh and wet- and dry-ground through 200 mesh. The ground products were cycloned, and the cyclone underflow constituted the flotation feed.

Flotation of Wet-Ground Minus 100-Mesh ROM Talc

The first flotation experiment on ROM talc was made on a sample that had been wet ground through 100 mesh and cyclone classified for removal of fines.

The 100-mesh grind, as a possible maximum size limit, was made for the following reasons:

- (1) Coarse platelets probably would exhibit a higher luster than fine platelets.
- (2) A 100-mesh grind is substantially less expensive than a finer grind.
- (3) A 100-mesh grind produces less fines to be rejected than a finer grind, and consequently the over-all yield or recovery would be greater.
- (4) It was desirable to know whether the froth product from a 100-mesh grind would be gritty, even though platy in structure.

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TABLE 4. FLOTATION RESULTS OBTAINED FROM ROM TALC WET-GROUND THROUGH 100 MESH

Product	Weight Per Cent	Platy	Nonplaty	Mineral Count, per cent			Reagents Added, lb./ton. of flotation feed		Feed, per cent solids
				Dolomite	Tremolite	HCl	Dowfroth 200		
<u>Test 88</u>									
Float-1	66.1	96	3	<1	1.42	0.05	9.3	--	
Float-2	22.9	95	4	<1	0	0.21	--	--	
Underflow	11.0	55	26	16	3	--	--	--	
Total	100.0	91	6	2-3	1-2	1.42	0.26		
<u>Test 89</u>									
Float-1	60.2	97	2	<1	0.0	0.05	10.0	--	
Float-2	20.5	Not evaluated			0.0	0.21	--	--	
Underflow	19.3	Not evaluated			--	--	--	--	
Total	100.0				0.0	0.26			

Note: Flotation feed was cyclone underflow, which represented 83.0 per cent of the weight of the original ground sample.

Tests 88 and 89 were duplicates, except that hydrochloric acid was used in Test 88 and no acid was used in Test 89. Table 4 shows the experimental conditions and the results.

The results given in Table 4 are not particularly encouraging, because neither experiment yielded a Float-1 product containing more than 97 per cent platy talc. The Float-1 product of Test 88 contained 96 per cent platy talc and Float-1 of Test 89 contained 97 per cent platy talc. The difference between 96 and 97 per cent is not considered significant, and the two tests can be considered to yield the same quality of Float-1 product. However, there was a marked difference in the amount of weight recovered in the Float-1 products. In Test 88, 1.42 pounds of HCl per ton were used, and the Float-1 product represented 66.1 per cent of the flotation feed weight. When no acid was used, as in Test 89, the weight per cent of Float-1 was 60.2 per cent. Prior experience with Italian No. 2 talc implied that the reverse condition would result. That is, higher recoveries usually result when no acid is used, although the platy content might decrease slightly. It would not be safe to call these tests conclusive as long as this anomaly is not confirmed.

Both Float-1 products had a high luster but felt gritty.

The Float-1 product of Test 88 was screened on 150- and 200-mesh Tyler sieves, and the separated fractions were examined by microscope. The results are given in Table 5.

TABLE 5. PROPERTIES OF FLOAT-1 PRODUCT FROM MINUS 100-MESH WET-GROUND TALC

Size, Tyler Mesh	Weight Per Cent	Platy Talc, per cent	Remarks
- 100+150	10.1	99	High luster, gritty
- 150+200	23.1	98-99	High luster, gritty
- 200	66.8	95	High luster, good slip
Total	100.0	96	High luster, gritty

Table 5 shows that the plus 200-mesh particles were 98 to 99 per cent platy talc, whereas the minus 200-mesh particles were only 95 per cent platy talc. Although the plus 200-mesh talc was of high purity, the thickness of the platelets produced a gritty texture. The minus 200-mesh portion, though only 95 per cent platy talc, had both a high luster and a good slip.

It is probable that the platy content of the Float-1 products from a 100-mesh grind could be improved; but, because of the objectionable gritty nature of the powder, it was decided to investigate the results obtainable from the conventional 200-mesh grind.

Flotation of Dry-Ground Minus 200-Mesh ROM Talc

Experiments were made on ROM talc that had been wet-ground and dry-ground through 200 mesh in a pebble mill. The ground products were cycloned for removal of the extreme fines, and the cyclone-underflow products were floated in the usual manner.

Three experiments were made on dry-ground talc and the results are given in Table 6.

The results given in Table 6 show that 97 per cent platy talc was obtained in the Float-1 product of each test. Tests 131 and 132 were made in an identical manner and, although the Float-1 products both contained 97 per cent platy talc, there was a noticeable difference in the weights recovered. Test 131, Float-1, contained 68.7 per cent of the flotation feed weight, but Test 132, Float-1, contained only 63.5 per cent of the flotation feed weight. There is no obvious explanation for this difference in weight recoveries. It is noticed, however, that the weight recovered in the Float-1 combined with the Float-2 is about the same, 91 to 92 per cent, in each of the three tests.

When the Float-1 products were examined under the microscope, it was found that some of the platelet surfaces were pitted. The amount of pitted platelets reporting to the Float-1 product was about 2 per cent, or 95 per cent normal platelets plus 2 per cent pitted platelets. This pitting or pockmarking was not noticed again either in the Italian No. 2 talc or in the subsequent ROM wet-pebble-milled flotation products, and apparently is a characteristic of dry-pebble milling.

Flotation of Wet-Ground Minus 200-Mesh ROM Talc

Flotation tests were made on wet-ground products, after cycloning for rejection of fines, to investigate the effect of acid strength and frother types on the Float-1 products.

Effect of HCL on Recovery and Quality. The effect of HCL on the flotation of Italian No. 2 talc was discussed briefly in the First Progress Report and in some detail in the Second Progress Report on "The Physical Concentration of Talc Ores". In the Second Progress Report, of July 31, 1959, it was stated in the "Summary" that, "Hydrochloric acid added in the

TABLE 6. RESULTS OBTAINED FROM FLOTATION OF DRY-GROUND, WET-CYCLONED ITALIAN ROM TALC

Product	Weight(a) Per Cent	Mineral Count, per cent			Reagents Added, lb/ton of fлотation feed			Pulp, PH Per Cent Solids	
		Platy	Nonplaty	Dolomite	Tremolite	HCl	Dowfroth 200	Dowfroth 250	
<u>Test 103(b)</u>									
Float-1	63.3	97	<2		1	0.00	0.07	0.00	8.6(c)
Float-2	27.7	Not evaluated				0.00	0.28	0.00	
Underflow	9.0	Not evaluated				--	--	--	
Total	100.0					0.00	0.35	0.00	
<u>Test 131(d)</u>									
Float-1	68.7	97	1	0.8	1	1.54	0.00	0.06	6.9(e)
Float-2	23.3	Not evaluated				0.00	0.00	0.23	6.9
Underflow	8.0	Not evaluated				--	--	--	
Total	100.0					1.54	0.00	0.29	
<u>Test 132(d)</u>									
Float-1	63.5	97	1	0.7	1	1.57	0.00	0.06	7.0(e)
Float-2	28.2	Not evaluated				0.00	0.00	0.25	7.0
Underflow	8.3	Not evaluated				--	--	--	
Total	100.0					1.57	0.00	0.31	

(a) Weight per cent refers to per cent of flotation feed.

(b) Flotation feed was treated in a 8.50-liter-capacity (nominal) Deco flotation cell.

Tests 131 and 132 were made in a 1.75-liter-capacity (nominal) Fagergren flotation feed.

(c) Distilled water was used to form the talc slurry.

(d) Tests 131 and 132 were intended to be duplicate tests.

(e) Deionized water was used to form the talc slurry.

correct quantity, between 1.13 and 2.30 pounds per ton of feed solids, was effective in maintaining the purity of finished talc at 97 to 98 per cent platy particles. This amount of acid created a pulp pH ranging between 6.9 and 7.8 during flotation." It was stated elsewhere in the report that the addition of HCl in amounts up to 2.30 pounds per ton of feed solids would appear to be justified only if it were effective in inhibiting the inclusion of fine talc and aiding in froth control.

Tests 121, 122, 123, and 124 were made to compare the results obtained when acid was used and when it was omitted. Dowfroth 200 was used as the collector-frother in each test. The results given in Table 7 show that, when HCl was used in the amount of 2.05 to 2.34 pounds per ton of flotation feed, the Float-1 product was 99 per cent platy talc. The amount of weight recovered in Float-1 was higher when acid was used than when it was omitted, although the weight recovered decreased when the strength of the acid was increased from 2.05 to 2.34 pounds per ton.

The data given in Table 7 are not fully consistent. In Tests 122 and 124, when no acid was used, the Float-1 products were 97 and 99 per cent platy talc, respectively, and a weight recovery of about 55 per cent of the flotation feed was obtained in each product. If the difference in quality is important, more experiments would be necessary to establish the cause of the difference in platy talc content.

In Test 121, 63.8 per cent of the flotation feed weight was recovered in Float-1 and the product was 99 per cent platy talc. The pH during flotation varied between 6.4 and 6.6. A size distribution, by sedimentation, showed that 19.7 per cent of the Float-1 product was finer than 10 microns. This could mean that the flotation feed had been incompletely classified (the original ore being overground) and that the amount of HCl added had not been highly effective for additional rejection of fine particles during flotation.

Effect of Type of Frother on Recovery and Quality. Two different frothers were investigated to determine the effect on the recovery and quality of the Float-1 products. These frothers were Dowfroth 200 and Dowfroth 250. Both of these frothers are 100 per cent water soluble, although Dowfroth 250 is classed as the stronger of the two. The manufacturer, Dow Chemical Company, Midland, Michigan, claims that less Dowfroth 250 is needed to accomplish the same effect as a larger amount of Dowfroth 200.

The results of experiments that illustrate the influence of the type of frother on flotation results are given in Table 8.

Data given in Table 8 show that, under certain operating conditions, Dowfroth 250 was a stronger frother-collector than Dowfroth 200.

TABLE 7. EFFECT OF HC1 ON FLOTATION RESULTS

Product	Weight(a) Per Cent	Mineral Count, per cent			Reagents Added, lb/ton of flotation feed			Pulp Per Cent	
		Platy	Nonplaty	Dolomite	Tremolite	HCl	Dowfroth	200	pH
<u>Test 121</u>									
Float-1(b)	63.8	99	<1	<1	<1	2.05	0.08	6.4	6.9
Float-2	19.1	95				0.00	0.31	6.6	
Underflow	17.1					--	--		
Total	100.0					2.05	--	0.39	
<u>Test 122</u>									
Float-1	55.6	97	1	1	<1	0.00	0.08	7.8	6.2
Float-2	27.5					0.00	0.34	6.9	
Underflow	16.9					--	--		
Total	100.0					0.00	0.42		
<u>Test 123</u>									
Float-1(b)	57.5	99	<1	0.4	<1	2.34	0.09	6.7	
Float-2	24.3					0.00	0.35	6.9	
Underflow	18.2					--	--		
Total	100.0					2.34	0.44		
<u>Test 124</u>									
Float-1	55.5	99	<1	0.5	<1	0.00	0.09	8.1	5.9
Float-2	25.4					0.00	0.37	7.0	
Underflow	19.1					--	--		
Total	100.0					0.00	0.46		

(a) Weight per cent refers to per cent of flotation feed.

(b) The Float-1 products of Tests 121 and 123 were given to W. H. Ashton, of Johnson and Johnson, and were considered representative of what may be expected from a pilot-plant operation. These products, although containing an excess amount of minus 10-micron particles, were of interest principally because of their high luster.

TABLE 8. EFFECT OF TYPE OF FROTHER ON FLOTATION RESULTS

Product	Weight(a) Per Cent	Mineral Count, per cent			Reagents Added, lb./ton of flotation feed			Pulp Per Cent Solids	
		Platy	Nonplaty	Dolomite	Tremolite	HCl	Dow 200	Dow 250	pH
Test 121									
Float-1	63.8	99	<1	<1	<1	2.05	0.08	0.00	6.4
Test 125									
Float-1	64.1	98	<1	0.3	1	2.06	0.00	0.08	6.5
Test 122									
Float-1	55.6	97	1	1	<1	0.00	0.08	0.00	7.8
Test 126									
Float-1	66.0	96	1	0.3	2	0.00	0.00	0.08	7.7
Test 157									
Float-1	51.8	97	2	0.3	<1	2.45	0.11	0.00	5.8
Test 158									
Float-1	56.3	97	2	0.3	<1	2.40	0.00	0.11	5.6
Tests 147-148									
Float-1	62.1	97	2	<1	<1	1.66	0.00	0.06	6.4
Test 142									
Float-1	55.9	96	<3	0.2	<1	2.72	0.00	0.09	5.4
									6.3

(a) Weight per cent refers to per cent of flotation feed.

A comparison of Tests 121 and 125 shows that, when about 2 pounds of HCl and 0.08 pound of either frother per ton of flotation feed was used (creating a pH of 6.4 to 6.5), the amount of weight recovered in the Float-1 product was approximately 64 per cent. The quality of the floated products was essentially the same at 98 to 99 per cent platy talc particles.

These experiments were repeated without acid, but the amount of frother added was kept at 0.08 pound per ton of flotation feed, and are reported as Tests 122 and 126. When Dowfroth 200 was used, the weight recovered from the flotation feed was 55.6 per cent, compared with 66.0 per cent when the stronger Dowfroth 250 was used. The platy content of the float product, however, was only 96 per cent when Dowfroth 250 was used, compared with 97 per cent when Dowfroth 200 was used.

The weight recovery of 55.6 per cent in the Float-1 product of Test 122 appears too low and probably should not be considered as a firm figure without repeating the experiment.

Tests 142, 157, and 158 were made with increased amounts of acid and frother. The amount of frother was increased to obtain higher recovery and the amount of acid was increased to retard the flotation of undesirable minerals. The results show that, when the acid strength was in excess of 2.40 pounds per ton of feed, it had a definite tendency to decrease the weight recovered, even though the amount of frother-collector was increased from 0.08 to 0.11 pound per ton of flotation feed. In addition to this, the increased acid strength was not effective in improving the platy content beyond 97 per cent.

The conclusions from the experiments reported in Table 8 are:

- (1) Maximum grade and recovery are effected adversely if the flotation-pulp pH is less than 6.4, regardless of which frother is used.
- (2) Dowfroth 250 is a stronger frother-collector for platy talc than Dowfroth 200.
- (3) Better quality float products can be obtained with acid than without, providing the pH does not become less than 6.4.

All Float-1 products from ROM talc had a high luster; that is, the luster from these products was definitely of a higher order than was obtained from any float products from Italian No. 2 talc.

The deionized water used in all flotation experiments from Test 121 through Test 158 had a resistance of 105,000 to 150,000 ohms per cubic centimeter.

The amount of time available did not permit an extensive evaluation of the physical properties of the flotation products, and for the most part the products were rated solely by a microscope mineral count and subjective measurements of luster and feel or slip. The flotation products obtained compared favorably with the Italian No. 2 flotation products with respect to platy talc and dolomite content, and therefore it was believed that the properties of lubricity and alkalinity (pH of moistened, beneficiated products) would be essentially the same for both the ROM and the Italian No. 2 talcs. Bulk density was spot-checked on various Float-1 products and found to be in the range of 23 to 25 pounds per cubic foot.

The amount of minus 10-micron particles contained in the Float-1 products was also spot-checked and found to be between 9 and 20 per cent. The appearance of any excess amount of minus 10-micron particles in the Float-1 products is attributed to overgrinding and incomplete cyclone classification, rather than to flotation. In fact, there is evidence that flotation is helpful in the rejection of minus 10-micron particles.

The weight recovery expected from a continuous operation can be estimated reasonably closely by using data available from Test 121 (see Table 7). In this test, the Float-1 product was 63.8 per cent of the flotation feed weight and the Float-2 product contained 19.1 per cent of the flotation feed weight. By returning the Float-2 product back to the new feed, it would be reasonable to expect that another 63.8 per cent of it would be recovered. Therefore, an estimated weight recovery from the flotation feed would show $63.8 + \frac{63.8 \times 19.1}{100} = 76$ per cent. These calculations do not include a potential additional recovery that may be expected from scavenging the flotation underflow. It would not be unreasonable to expect an additional 3 to 5 per cent recovery by the scavenging step, followed by returning the scavenger froth back to the new feed. On this basis the total projected recovery in a continuous operation would be 80 per cent of the weight of the flotation feed.

The over-all recovery of high-grade talc from the original ore is also related to the efficiency of the pebble milling and hydraulic cycloning. Ore preparation as followed in Test 121 shows that 48.5 per cent of the original weight was rejected in the cyclone overflow as approximately minus 10-micron particles. This amount, 48.5 per cent, is far too much weight loss and should not be considered realistic, because the talc was overground. Ore preparation as followed in Test 151 (see Appendix) showed that only 31.7 per cent of the weight was rejected as cyclone overflow. This amount is also believed to be greater than would be obtained

from a continuous pilot mill or commercial circuit; 30 per cent is a realistic amount for estimating purposes. Therefore, the over-all estimated weight recovery would be $\frac{70 \times 80}{100} = 56$ per cent of the weight of the original ore.

Not all of the flotation experiments on Italian ROM talc are discussed in the text of this report. A complete tabulation of the experiments, showing the pertinent data, is presented in the Appendix.

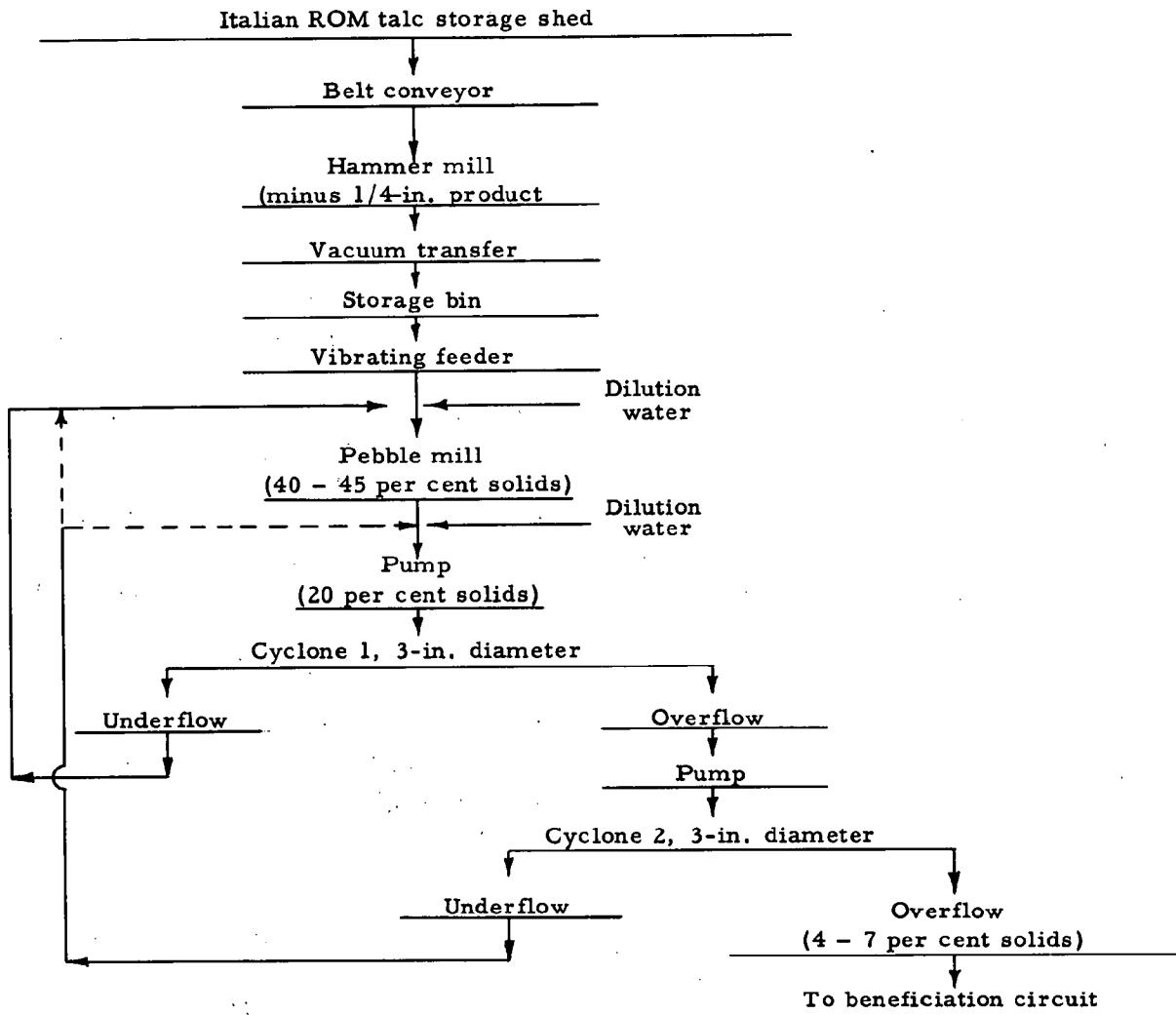
PROPOSED PILOT-PLANT FLOWSHEET

Figures 6 and 7 show the proposed flowsheets based on laboratory experiments for crushing, grinding, and beneficiation of Italian ROM talc.

The crushing circuit is to be operated intermittently, to replenish the storage bin, but the grinding circuit is a continuous operation, so that the beneficiation process will have uninterrupted feed. The objective of the flowsheet design shown in Figure 6 is to provide a flexible system for treating a variety of ROM talc ores (not specifically Italian). This circuit is expected to handle talc that may be received in pieces as large as 8- or 10-inch slabs, hammermill in one pass through 1/4 inch, wet grind, and classify at approximately 200 mesh. The minus 200-mesh pulp is expected to leave the circuit at 4 to 7 per cent solids, which is an ideal feed to the beneficiation circuit. It is expected that the amount of minus 10-micron talc produced can be held to a reasonable maximum by controlling the circulating load in the grinding circuit, grinding pulp density, pebble diameter, and total weight of charge, and, finally, by dilution of cyclone feed.

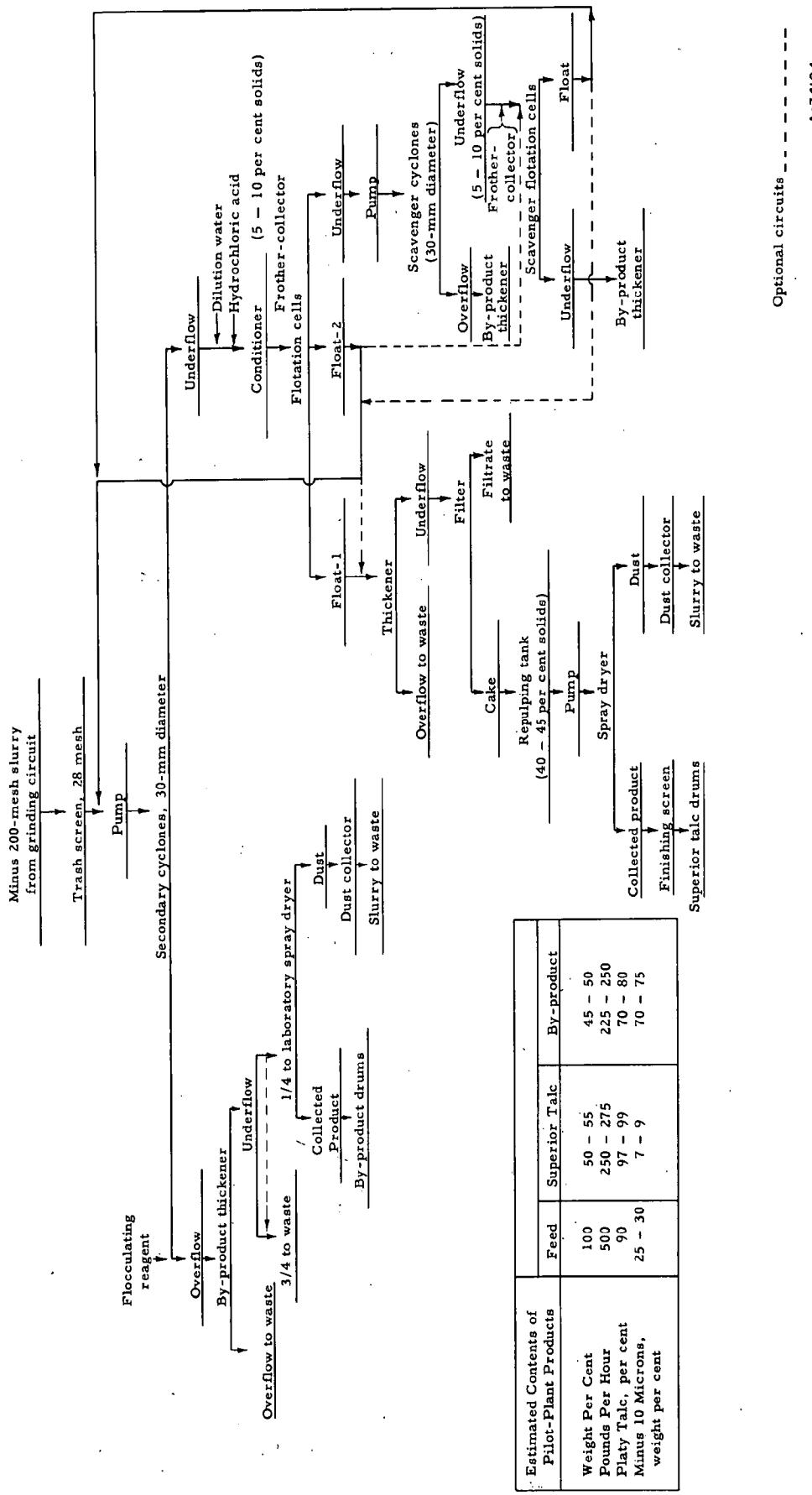
The flowsheet shown in Figure 7 is essentially the same as that developed for processing the Italian No. 2 talc*. Laboratory experiments showed that the same general results would be obtained from either ROM or Italian No. 2 talc. If the grinding circuit could be operated efficiently, a higher yield of beneficiated talc could be expected from the ROM talc than from the Italian No. 2 talc.

*Brown, W. E., "The Physical Concentration of Talc Ores - Flotation of Italian No. 2 Talc", Battelle Progress Report to Johnson and Johnson (July 31, 1959).



A-34125

FIGURE 6. PROPOSED PILOT-PLANT FLOWSHEET -
CRUSHING AND GRINDING CIRCUIT



B A T T E L L E M E M O R I A L I N S T I T U T E

FIGURE 7. PROPOSED PILOT-PLANT FLOWSHEET - BENEFICIATION CIRCUIT

A-34124

CONCLUSIONS

Data and observations obtained from the grinding, cycloning, and flotation experiments have established that:

- (1) Italian ROM talc can be beneficiated by the combined processes of crushing, grinding, classifying, and flotation. The finished beneficiated talc will have a high luster and will be 97 to 99 per cent platy talc. The yield expected in a continuous operation is 80 per cent of the flotation feed, or 56 per cent of the original ore.
- (2) Wet-pebble-mill grinding is more effective in obtaining 200-mesh grinds than dry-pebble-mill grinding, although less minus 10-micron talc is produced from dry grinding.
- (3) Wet-pebble-mill grinding yields a product that, after cycloning and floating, shows a marked improvement in luster compared with the products obtained from the beneficiation of Italian No. 2 talc.
- (4) ROM talc must be ground finer than 100 mesh, or the resulting flotation products will have a gritty texture.
- (5) The highest quality products were obtained when the flotation feed pulp was maintained at a pH of 6.4 or higher.
- (6) Water-soluble frothers, such as Dowfroth 200 or Dowfroth 250, are good promoters for the flotation of platy talc. There are indications that Dowfroth 250 is the stronger promoter of the two, but it may be slightly less selective.
- (7) The experimental results show that the processes of classification, flotation, and filtering as developed for Italian No. 2 talc can be adapted to Italian ROM talc. No change in equipment types or sizes should be necessary.

Table 9 shows the Johnson and Johnson specifications for Italian No. 1 talc and includes a comparison of these specifications with the beneficiated products from the Italian No. 2 and ROM talcs.

This table shows that the beneficiated products contain less than 0.75 per cent of dolomite and less than 1 to 3 per cent of nonplaty minerals. The bulk density of the beneficiated product was within the specification of 22 to 27 pounds per cubic foot.

TABLE 9. COMPARISON OF SPECIFICATIONS WITH THE BENEFICIATED PRODUCTS FROM
ITALIAN NO. 2 AND ROM TALCS

Physical-Property Control	Specification	Italian No. 1, Johnson and Johnson Raw Material	Italian No. 2, Laboratory Beneficiated	Italian ROM, Laboratory Beneficiated
Moisture ^(a) , per cent	<0. 15	0. 05	<0. 05	<0. 05
Solubility in HCl, per cent	<6	2. 1-2. 8	<0. 75 ^(b)	0. 3-0. 6 ^(b)
Fineness, per cent through 200 mesh	Not less than 98. 5	99. 8	99. 5	98. 5
Bulk density, lb./ft. ³	Not less than 2.2 nor more than 2.7	23. 0	28-29	23-25
Microscopic structure, per cent platy	Platelet showing no acicular or excessive granular crystals	88-90	97-99	97-99
pH (Alkalinity)	7. 0-7. 5	9. 0-9. 3	8. 1-8. 8	8. 3-8. 6

(a) Moisture content would be significant only from a continuous plant operation, because laboratory products can be dried to any desired moisture content.

(b) Solubility in HCl as reported here is expressed as dolomite content, which was determined from CO₂ assay.

FUTURE WORK

Since the completion of the experimental work included in this report, a new reagent combination, involving Aerosol, has been developed. The development has led to improved results and will be presented in a separate report.

An experimental program is now in progress to establish how much of the water used in beneficiation can be re-used without adverse results to the over-all process.

The original notes on the laboratory work described in this report are in Battelle Laboratory Record Books 14668, 15042, 15190, 15456, and 15662. The work was done in the period from July 24, 1958, to April 20, 1959.

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APPENDIX

SUMMARIZED RESULTS OF ALL FLOTATION TESTS MADE ON
ITALIAN ROM TALC

B A T T E L L E M E M O R I A L I N S T I T U T E

Test	Initial Weight, grams	Grind Interval, minutes	Time, minutes	Preparation		Cyclone Product	Flotation		Mineral Count, per cent	Per Cent Solids in water	PH of HCl(a)	Dorffit 200	Dorffit 250	Remarks	
				Feed, psi	Overflow, weight per cent		Weight Per Cent of Original Feed	Weight Per Cent of Flotation Feed	Platy Nonplaty	Dolomite	Tremolite	Pulping			
88	430	15	14.7	17.0	83.0	Float-1	56.1	54.9	3 <1	<1	<1	Distilled N.D.(b)	9.3	1.42 0.05	
				Float-2	22.9	19.0	95	4	<1	<1	<1	Distilled N.D.(b)	0	0.21	
89	430	15	14.7	17.0	83.0	Underflow	11.0	9.1	35	26	16	Distilled N.D.(b)	3	0.05	
				Float-1	60.2	50.0	97	3	<1	<1	<1	Distilled N.D.(b)	10.0	0.05	
90	510	60	14.7	26.9	73.1	Float-2	20.5	17.0	95	4	<1	Distilled N.D.(b)	0	0.21	
				Float-1	63.5	46.4	95	1	Trace	Trace	Distilled	8.3	0.07		
91	510	60	14.7	26.9	73.1	Float-2	16.1	11.8	97	3	<1	Distilled N.D.(b)	0	0.26	
				Float-3	14.7	10.7	95	4	<1	<1	<1	Distilled N.D.(b)	0	0.65	
				Cleaner Float	45.8	35.7	99	<1	<1	<1	<1	Distilled N.D.(b)	8.0	0.07	
				Float-2	32.0	23.4	96	3	<1	<1	<1	Distilled N.D.(b)	0	0.92	
				Cleaner U.F. off	10.8	7.9									
				Underflow	8.4	6.1									
92															
93															
94	510	60	14.7	29.7	70.3	Float-1	67.3	47.3	98	1	0.4	<1	Distilled N.D.(b)	4.5	0 0.13
				Float-2	22.7	16.0			97	<1	1	Distilled N.D.(b)	4.8	0 0.39	
103	500	60	14.7	Not determined		Float-1	63.3					Distilled N.D.(b)	0	0 0.07	
				Float-2	27.7						Distilled N.D.(b)	0	0 0.28		
104															
121	120	15	14.7	48.5	51.5	Discarded. Results erratic because of overgrinding.	63.8	32.9	99	<1	<1	<1	Deionized N.D.(b)	6.9	2.05 0.08
				Float-1	19.1	9.8						Deionized N.D.(b)	0	0 0.31	
122	120	15	14.7	48.5	51.5	Discarded because of contamination. Products used for filtration experiments.	55.6	28.6	97	1	1	<1	Deionized N.D.(b)	6.2	0 0.08
				Float-2	27.5	14.2						Deionized N.D.(b)	0	0 0.34	
123	120	15	14.7	50.7	49.3	Float-1	57.5	28.3	99	<1	0.4	<1	Deionized N.D.(b)	6.1	2.34 0.09
				Float-2	24.3	12.0						Deionized N.D.(b)	0	0 0.35	
124	120	15	14.7	50.7	49.3	Float-1	55.5	27.4	99	<1	0.5	<1	Deionized N.D.(b)	5.9	0 0.09
				Float-2	25.4	12.5						Deionized N.D.(b)	0	0 0.37	
125	120	15	23.0	46.8	53.2	Float-1	64.1	34.1	98	<1	0.3	1	Deionized N.D.(b)	6.9	2.06 0.08
				Float-2	23.4	12.4						Deionized N.D.(b)	0	0 0.32	
126	120	15	23.0	46.8	53.2	Float-1	66.0	35.1	96	1	0.3	2	Deionized N.D.(b)	6.8	0 0.33
				Float-2	21.1	11.2						Deionized N.D.(b)	0	0 0.33	
129	240	11	23.0	34.7	65.3	Float-1	65.6	42.8	97	2	0.3	<1	Deionized N.D.(b)	7.5	1.90 0.08
				Float-2	21.2	13.8						Deionized N.D.(b)	0	0 0.30	
130	240	11	23.0	34.7	65.3	Float-1	64.0	41.8	97	2	0.3	<1	Deionized N.D.(b)	7.3	2.06 0.08
				Float-2	24.0	15.7						Deionized N.D.(b)	0	0 0.31	
131	120	15	23.0	25.2	74.8	Float-1	68.7	51.4	97	1	0.8	1	Deionized N.D.(b)	9.7	1.54 0.08
				Float-2	23.3	17.4						Deionized N.D.(b)	0	0 0.23	
132	120	15	23.0	25.2	74.8	Float-1	63.5	47.5	97	1	0.7	<1	Deionized N.D.(b)	8.9	1.57 0.06
				Float-2	28.2	21.1						Deionized N.D.(b)	0	0 0.25	
141	240	11	14.7	36.7	63.3	Float-1	56.5	35.8	97	<3	0.2	<1	Deionized N.D.(b)	6.7	2.59 0.08
				Float-2	27.7	17.5						Deionized N.D.(b)	0	0 0.34	
142	240	11	14.7	36.7	63.3	Float-1	55.9	35.4	96	<3	0.2	<1	Deionized N.D.(b)	6.3	2.72 0.09
				Float-2	26.9	17.0						Deionized N.D.(b)	0	0 0.36	
147, 148	240	11	23.0	35.7	64.3	Float-1	62.1	39.9	97	2	<1	<1	Deionized N.D.(b)	7.9	1.66 0.06
				Float-2	23.3	15.0						Deionized N.D.(b)	0	0 0.27	
151	240	11	23.0	31.7	68.3	Float-1	54.6	37.4	96	2	<1	1	Deionized N.D.(b)	6.3	2.05 0.05
				Float-2	32.9	22.5						Deionized N.D.(b)	0	0 0.41	
152	240	11	23.0	31.7	68.3	Float-1	47.8	32.6	97	2	<1	<1	Deionized N.D.(b)	6.3	0 0.41
				Float-2	36.8	23.9						Deionized N.D.(b)	0	0 0.41	
157	240	11	23.0	40.7	59.3	Float-1	51.8	30.7	97	2	0.3	<1	Deionized N.D.(b)	5.3	2.45 0.11
				Float-2	25.5	15.1						Deionized N.D.(b)	0	0 0.46	
158	240	11	23.0	40.7	59.3	Float-1	56.3	33.4	97	2	0.3	<1	Deionized N.D.(b)	5.4	2.40 0.11
				Float-2	27.8	16.5						Deionized N.D.(b)	0	0 0.42	

(a) HCl is reported in terms of 35.5 to 38 per cent HCl having a specific gravity of 1.105 at 60° F.

(b) R.D. = not determined.

Note: Not all Float-2 products were examined for mineral distribution. Ordinarily, in a continuous operation, these products would show about 93 to 95 per cent clay talc and would be returned to the flotation circuit for further cleaning.

Exhibit 28

Battelle Memorial Institute

505 KING AVENUE COLUMBUS I. OHIO

January 24, 1968

Dr. W. H. Lycan
Director of Research
Johnson and Johnson
New Brunswick, New Jersey

Dear Dr. Lycan:

This letter report summarizes the results obtained to date on the beneficiation of talc by flotation which is a part of Phase 3 of our talc research program.

The primary objective of the flotation experiments was to obtain a product which was predominantly platy talc and which would contain a minimum of tremolite and carbonates. Only secondary attention was given to talc recovery.

Samples from three talc-bearing deposits were investigated. The mineralogical composition of these samples is given in Table 1. The composition of Italian Talc No. 1 is included for comparison.

TABLE 1. MINERALOGICAL COMPOSITION OF SAMPLES INVESTIGATED

Sample	Mineral Content, per cent			
	Platy	Nephty	Carbonates	Tremolite
Oasis Mine (Nevada)	48	43	5	4
Stone Creek Mine (Montana)	30	67	1	2
Italian No. 2	90	6	3	1
Italian No. 1	88-90	8-10	2	Trace

The three samples shown in Table 1 represent talc types containing low, intermediate, and relatively high percentages of platy talc. The Oasis and Stone Creek mine samples were tested for academic purposes and do not necessarily represent recommended uses. The sample identified as Italian No. 2 may be of more immediate interest to Johnson and Johnson.

Ten flotation tests were made and the results are summarized in Table 2.

TABLE 2. SUMMARIZED RESULTS OF TALC FLOTATION TESTS

Sample	Test No.	Product	Weight Per Cent	Approximate Mineral Count, Per Cent				Reagents Used
				Platy	Nonplaty	Carbonate	Tremolite	
Oasis		Head Sample	100.0	48	43	3	4	—
Oasis	1	Fleet 1	32.2	77	20	1	3	None
Oasis	2	Fleet 1	32.4	75	23	1	1	None
Oasis	3	Cleaner Fleet	31.6	50	18	1	1	Dowfroth 200
Oasis	5A	Fleet 1	34.6	53	16	Q	1	Dextrine
Oasis	8	Fleet 1	26.9	82	15	Q	2	Dextrine
Oasis	9	Fleet 1	34.6	77	17	4	2	None
Stone Creek		Head Sample	100.0	30	67	Q1	2	—
Stone Creek	4	Fleet 1	29.9	55	12	1	2	None
Stone Creek	6	Fleet 1	25.0	56	10	2	2	Dextrine, Na_2SiO_3
Italian No. 2		Head Sample	100.0	90	5	3	2	—
Italian No. 2	7	Fleet	76.9	95	3	Q	Q	Dextrine, Dowfroth 200
Italian No. 2	10	Fleet	75.3	95	3	Q	Q	Dextrine, Dowfroth 200
Italian No. 1*		Head Sample	100.0	85-90	8-10	Q2	Trace	

* Italian No. 1 talc is included in this table for comparison with the flotation products.

Battelle Memorial Institute

Dr. W. H. Lycan

Page

January 24, 1958

The data given in Table 2 show that it is relatively easy to obtain, from any of the samples tested, a flotation product enriched in platy talc. The Oasis head sample contained 48 per cent platy talc. The flotation product, Fleet 1, contained 83 per cent platy talc and represented 34.6 per cent of the flotation feed weight. The Stone Creek head sample contained 30 per cent platy talc. The flotation product, Fleet 1, contained 85 per cent platy talc and represented 29.9 per cent of the flotation feed weight.

Two flotation tests were made on Italian talc No. 2. The sample as received at Battelle ~~was~~ 90 per cent platy talc and 6 per cent non-platy talc. Flotation yielded a product containing 96 per cent platy talc and only 3 per cent of nonplaty talc. However, only 76.9 per cent of the original weight of the feed was recovered.

It is unlikely that the optimum results for purity of product and recovery of the desired platy talc were obtained. Further improvement in purity, however, is probably unnecessary but improved recovery is probably essential.

Flotation Test 8 (Oasis sample) was arbitrarily selected for a study of the mineral distribution in all products of a test. The results of this study are given in Table 3.

TABLE 3. FLOTATION RESULTS SHOWING MINERAL CONTENT IN ALL PRODUCTS OF TEST 8 (OASIS SAMPLE)

Product	Weight Per Cent	Approximate Mineral Content, per cent			
		Platy	Nonplaty	Carbonate	Tremolite
Fleet 1	26.9	82	18	1	2
Cleaner Fleet	42.1	60	38	1	4
Cleaner Underflow	14.2	24	75	1	2
First Underflow	16.8	25	75	16	0
Composite*	100.0	95	25	4	3
Head Sample		48	42	5	4

* A close material balance was not obtained and for this reason the calculated distribution of the minerals is not included in this table. The reason for the lack of a satisfactory balance is probably due to the fact that the proper weight relationships cannot be assigned to the different minerals unless they are closely sized. It will be necessary to develop a systematic and rapid evaluating procedure if an accurate balance is required. The ultimate evaluation of the products will be more pronounced by measurement of their physical properties.

Battelle Memorial Institute

Dr. W. H. Lyman

Page

4

January 24, 1958

An examination of the test products indicates that platy talc is selectively floated and a mineral count shows that the Fleet 1 product is 82 per cent platy talc. The carbonates are easily rejected and do not exceed one per cent of any of the test products except in the first underflow which contains 16 per cent carbonates. Tremolite and carbonates report predominantly in the first underflow; however, the rejection of tremolite is not as complete as the rejection of the carbonates.

After the encouraging flotation results were observed, it seemed necessary to establish that the enrichment of platy talc was the result of true flotation rather than a particle sizing effect. Samples of the flotation feed and the Fleet 1 product from the Oasis sample were screened on 200 mesh and the plus and minus 200-mesh fractions were evaluated with the microscope. The information obtained by this procedure is given in Table 4.

TABLE 4. MINERAL DISTRIBUTION IN SIZED FRACTIONS OF FLotation FEED AND FLEET 1 PRODUCT OF TEST 8 (OASIS SAMPLE)

Product	Mesh, size	Weight Per Cent	Mineral Count, per cent			
			Platy Talc	Noplasy Talc	Carbonates	Tremolite
Flotation Feed	-45+200	30	45	48	3	5
	-200	70	50	43	4	3
Fleet 1	-45+200	29	61	36	1	2
	-200	71	87	10	1	2

The data in Table 4 show clearly that the Fleet 1 product is improved in platy talc content in both size fractions. The plus 200-mesh fraction has been increased from 45 per cent platy talc to 61 per cent platy talc. The minus 200-mesh portion shows a significant improvement and has been increased from 50 per cent platy talc to 87 per cent platy talc. Because the grade improvement of the minus 200-mesh portion is so marked, it is implied that better flotation results might be obtained by grinding all of the feed through 200 mesh before flotation.

Two duplicate tests were made on Italian No. 2 talc which is 99 per cent minus 200 mesh. The float product of Test 7 represented 76.9 per cent of the feed and microscopic examination indicated that it was at least 96 per cent platy talc and about 3 per cent fibrous talc. The carbonate content was reduced from three per cent to less than one per cent and the tremolite content was less than one per cent.

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Dr. W. H. Lycan

Page 5

January 24, 1958

A comparison of Italian No. 1 talc (not beneficiated) with the
Float 1 products obtained from the other samples shows the following
mineral relationships:

Per Cent Weight Recovered	Mineral Content per cent				Tremolite
	Platy Talc	Nonplaty Talc	Carbonates	Tremolite	
Italian No. 1	100.0	88-90	2-10	<1	Trace
Italian No. 2 (Float 1)	76.9	91	3	1	Trace
Stone Creek (Float 1)	29.9	85	12	1	2
Oasis (Float 1)	24.6	83	16	<1	1

It is noted in the foregoing data that the Float 1 product obtained from the Italian No. 2 sample is better than any of the other products relative to degree of platineness. The Stone Creek and Oasis products are not as pure as Italian No. 1 talc.

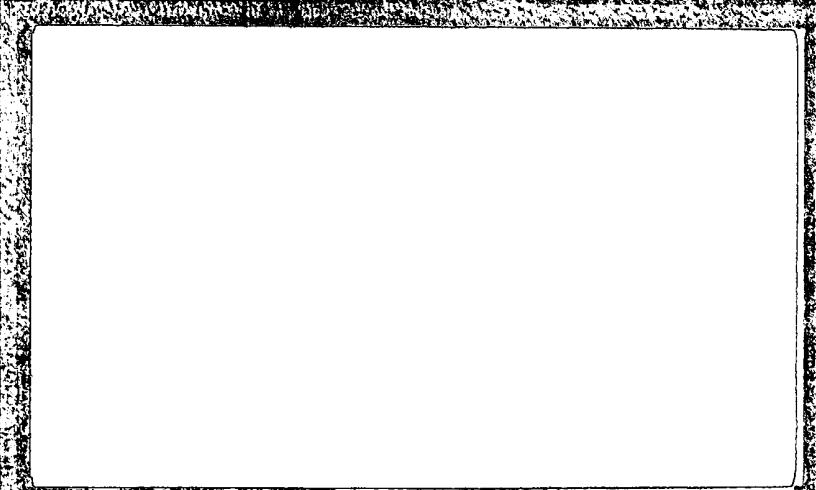
Beneficiation tests in the near future will be directed toward obtaining a higher recovery, without sacrificing quality, from the Italian No. 2 sample. Preliminary attention will be given to the filtering and drying characteristics of the enriched flotation products. Physical measurements will be made on the flotation products. Some attention will be given toward establishing a more accurate method of mineralogical evaluation.

Very truly yours,

Whitman E. Brown

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Exhibit 29



BATTELLE FIELDS OF RESEARCH

AERONAUTICAL ENGINEERING	INSTRUMENTATION
AGRICULTURAL SCIENCE	LIGHT ALLOYS AND RARE METALS
AIR AND STREAM POLLUTION CONTROL	MACHINERY ENGINEERING
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BIOCHEMISTRY	MINERAL PROCESSING
BIOPHYSICS	NONDESTRUCTIVE INSPECTION
CERAMICS	NUCLEAR ENERGY
CHEMICAL ENGINEERING	OPERATIONS RESEARCH
CORROSION TECHNOLOGY	ORGANIC CHEMISTRY
ECONOMICS	ORGANIC COATINGS
ELECTRICAL ENGINEERING	PETROLEUM ENGINEERING
ELECTROCHEMICAL ENGINEERING	PHYSICAL CHEMISTRY
ELECTROCHEMISTRY	PROCESS METALLURGY
ELECTRONICS	PRODUCTION ENGINEERING
EXTRACTIVE METALLURGY	PULP AND PAPER
FOOD AND FOOD PROCESSING	RADIOISOTOPES AND RADIATION
FOREST PRODUCTS	REACTOR TECHNOLOGY
FOUNDRY PRACTICE	RUBBER AND PLASTICS
FUELS AND COMBUSTION	SOLID-STATE DEVICES
GRAPHIC ARTS TECHNOLOGY	SYSTEMS ENGINEERING
HIGH-TEMPERATURE METALLURGY	TEXTILES AND FIBERS
INDUSTRIAL PHYSICS	THEORETICAL AND APPLIED MECHANICS
INFORMATION PROCESSING	TERMODYNAMICS
INORGANIC CHEMISTRY	WELDING TECHNOLOGY

PROGRESS REPORT

on

FURTHER STUDIES ON THE MEASUREMENT
AND CORRELATION OF THE PHYSICAL
PROPERTIES OF TALC

to

JOHNSON AND JOHNSON
Russell copy
May 9, 1958

by

W. L. Smith

BATTTELLE MEMORIAL INSTITUTE
505 King Avenue
Columbus 1, Ohio

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cc: W. L. Smith (3)

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R. L. Anderson

Battelle Memorial Institute

505 KING AVENUE COLUMBUS I, OHIO

July 18, 1958

Dr. W. H. Lycan
Director of Research
Johnson and Johnson
New Brunswick, New Jersey

Dear Dr. Lycan:

This letter transmits six copies of our report "Further Studies on the Measurement and Correlation of the Physical Properties of Talc".

This report, plus that of October 25, 1957, demonstrates that lubricity may be improved and abrasiveness lessened by the removal of the mineral contaminants from talc. A minimum number of physical-property measurements are recommended as important in the comparison of high-grade talcs and in the evaluation of improvement of physical properties through beneficiation.

Because of urgency on other phases of Battelle's investigation of talc, further studies on the physical properties are postponed.

We would be pleased to have your comments on our findings.

Very truly yours,

Wm. L. Smith
Principal Geologist
Minerals Beneficiation Division

WLS/djo
Enc. (6)

DEDICATED TO THE ADVANCEMENT OF SCIENCE

TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	1
INTRODUCTION AND SUMMARY	1
DISCUSSION OF ABRASIVENESS	3
THE MEASUREMENT OF ABRASIVENESS	5
Discussion	5
The Abrasion Machine	6
The Abrasiveness of Talc Samples	12
THE RELATIONSHIP OF ABRASIVENESS TO PARTICLE-SIZE DISTRIBUTION AND CONTAMINATION.	14
Discussion	14
Correlation of Abrasiveness With Particle-Size Distribution and Contamination.	14
MEASUREMENT AND CORRELATION OF OTHER PHYSICAL PROPERTIES . .	18
Moisture Content	18
Absorptive Power	20
Alkalinity	21
Acid Solubility	22
REFLECTANCE AND WHITENESS	23
Discussion	23
The Measurement of Reflectance and Whiteness	27
THE DUST COMPONENT	28
APPRAISAL OF PHYSICAL-PROPERTY MEASUREMENTS IN THE EVALUATION OF ORES AND BENEFICIATION PRODUCTS	30
FUTURE WORK	33
REFERENCES	33

APPENDIX A

DESCRIPTION OF ABRASION MACHINE AND TECHNIQUE OF OPERATION ..	A-1
---	-----

APPENDIX B

DETERMINATION OF EQUIVALENT DOLOMITE CONTENT IN ITALIAN TALC BY VOLUMETRIC ANALYSIS.	B-1
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BATTELLE MEMORIAL INSTITUTE

FURTHER STUDIES ON THE MEASUREMENT
AND CORRELATION OF THE PHYSICAL
PROPERTIES OF TALC

by

W. L. Smith

ABSTRACT

To establish the interrelationships of the physical properties of talc and to be able to visualize the means of their improvement, it has been necessary to devise means of measuring small differences in properties. To determine the nature and effects of grit, a chemical analysis for small concentrations of carbonate minerals and a machine for measuring the relative abrasiveness of talc samples were contrived, and the measurements were compared with those of other physical properties. The various other measurements were made on standard laboratory instruments, using both sized fractions and whole powder. Physical-property measurements of the talc demonstrated that the samples which produced the least abrasion were those with the greater platy talc component and those with the least amount of contaminants. It is concluded that the improvement of slip and the lessening of abrasiveness may be accomplished by the removal of the mineral contaminants, but not by the removal of size fractions.

Preliminary work on color and reflectance properties is presented, and demonstrates a relationship to particle size and, hence, secondarily to other physical properties.

The report includes an appraisal of the various physical-property measurements employed in the evaluation of improvement of talcs. A minimum number of measurements are recommended as important in the consideration of beneficiation for improvement and for comparison of natural high-grade ores.

INTRODUCTION AND SUMMARY

This report is a continuation of the studies of the physical properties of talc, their measurement, and comparison^{(1)*}, previously reported to Johnson and Johnson. The first Progress Report dealt with petrography, lubricity, and such physical measurements as average diameter, bulk density, porosity, and surface area. It was concluded from the previous study that the acceptable Italian talc fell within a small range of physical measurements and that the samples with the more desirable slip have the greater surface area, the smaller average particle diameter, the greater ratio of voids to total volume, and the lesser bulk density. Lubricity was found to be controlled by the shape of the relatively small content of larger particles in an otherwise finer mixture. Removal of the coarser contaminants, or preferably of all of the contaminants, was concluded to be a means of improving the slip of the talc.

* References appear at end of report.

The conclusions of the previous progress report have been further substantiated by the following studies; however, since lubricity is but one of many of the properties of talc, the previous work represented but part of the picture. Whereas the previous report dealt primarily with the physical measurement of areas, diameters, weight, and directly related characteristics, this report deals with reflectance, color, moisture content, abrasiveness, alkalinity, and acid solubility - properties related to lubricity only through their common correlation through surface area, size, and component contamination.

As in the previous report, both particle size and shape and the amount and nature of the contaminants are investigated to determine the contributing factors to physical measurement variations. It is recommended as a result of these studies that the following measurements are sufficient to determine satisfactory talc within the range in composition of Italian talc. These are the determination of the mineralogy and particle size distribution, volumetric analysis of the carbonate component, and the measurements of the bulk density, moisture content, reflectance, and whiteness. This should also serve as a basis for the determination of acceptability in other talcs and beneficiated products, taking into consideration the differences in size distribution, crystallographic habit, and mineral contamination.

Because size distribution, as it is reported, is dependent upon the analytical procedure, the measurement of physical properties is made only on closely sized fractions in the coarser size ranges. Division into size fractions of the finer size portion of a powder which is composed of platelets is inaccurate by standard laboratory procedures and requires detailed petrographic examination of the products. A method proposed by R. W. Schatz⁽²⁾ which reports size distribution on the basis of theoretical spheres rather than on the basis of actual petrographic measurement serves as an excellent comparative measurement for powders with similar mineralogical and crystallographic composition. Of necessity, these figures show little resemblance to the measurements of the greater dimensions of the talc platelets. Consistent with the previous progress report, the particle-size-distribution data here presented are based on screened size fractions and the sizes given are those measured on a petrographic microscope.

As in the study of lubricity, in order to determine the improvement of specific physical properties, objective tests had to be devised to measure the small differences between acceptable talc and talc of lower quality. Until now, acid solubility was determined gravimetrically on the various talc samples. The small differences in per cent composition and per cent incidence of the carbonate component of Italian talc, and its close relationship to both abrasiveness and lubricity, required the development of an analysis for equivalent dolomite in low concentrations (Appendix B).

Abrasiveness has previously been measured subjectively, similar to lubricity. However, because subjective measurements are not correlative, and because small, often significant differences cannot be so measured, an abrasion machine was built. This device, does not give an absolute value to abrasion, however, it provides reproducible figures which are of relative value and which are correlative with measurements of other physical properties.

Except where otherwise noted, the measurements presented in this report were made on the same samples of "EGT Extra 00000" talc, obtained from the Cranford, New Jersey, plant, which were used in the work reported in the previous Progress Report on physical properties.

DISCUSSION OF ABRASIVENESS

A highly undesirable property of a talcum powder is abrasiveness or grittiness. Grit is undesirable in that it may scratch or otherwise irritate the skin, and even very small amounts of grit may quickly be noticed subjectively.

Grit consists of that portion of ground talc which is angular, or oversize, particularly in thickness. Grit includes both oversize and nonplaty talc particles as well as mineral contaminants. It occurs as aggregates of talc and contaminants, as acicular and fibrous particles of talc and amphibole, as shards and granules of amphibole or carbonate, and as prismatic grains of titanite, rutile, zircon, apatite, and other accessory minerals.

Where the grit is other than oversize talc, it has hardness and angularity sufficient to scratch. Talc which is oversize in its greater dimensions is rare in the samples studied. It is the product of incomplete grinding and may easily be removed on a 150-mesh screen. Talc which is oversize in thickness is of the nonplaty variety, the result of the incomplete alteration of pre-existing minerals or the formation of pseudomorphs after more equidimensional species. Such particles serve less as abrasives than as deterrents to proper slip. The 8 to 10 per cent of nonplaty talc in the Italian material is presumed to be derived from tremolite or enstatite. This mechanism is discussed in the reports on the Brazilian⁽³⁾ and Canadian⁽⁴⁾ talc deposits.

Whereas friction, as expressed in the sense of the translation movements of talc platelets over one another, produces the desirable property of slip, such friction is not a disruption of the free lamellar movement of the component particles of the powder nor a disruption of the free movement of the surfaces in contact. When, however, a lubricant fails to mask irregularities in the contacting surfaces or introduces asperities of its own, then point friction or plowing is initiated. Point friction and plowing are the sources of irritation or grittiness. Grit permits wear between the contacting surfaces by abrasion, either in the plowing or scratching mechanism of oversize and angular particles, or by the disruption of lamellar movement of the platelets which leaves areas unlubricated or introduces a damming-up and rolling of particles.

Although lubricity and abrasiveness may seem to be relative, or the presence of one may seem to preclude the presence of the other, no direct correlation should be expected between the two properties inasmuch as both are the functions of several variable factors. A decrease in grit, however, is certain to improve the lubricity of whole powders where particle size is not a controlling factor.

Idealized talc particles are rounded platelets which may be thought of as essentially two dimensional, the thickness being about 1/8 to 1/15 of the greater dimension, depending upon the crystalline nature of the mineral and the degree of subdivision attained. In the better grades of talc the greater dimensions of a platelet are nearly equal.

The Italian No. 1 talc contains from less than 1 per cent to about 3 per cent of contaminants. The contamination is natural and consists mostly of carbonate with minor amphibole and rare accessory minerals. The carbonate component has been identified petrographically as primarily dolomite ($\text{CaO} \cdot \text{MgO} \cdot 2\text{CO}_2$) plus a minor amount of probable magnesite ($\text{MgO} \cdot \text{CO}_2$). No calcite ($\text{CaO} \cdot \text{CO}_2$) was identified. The amphibole component has been established to be the variety tremolite ($2\text{CaO} \cdot 5\text{MgO} \cdot 8\text{SiO}_2 \cdot \text{H}_2\text{O}$).

Table 1 based on Table 2 of the previous Progress Report⁽¹⁾ lists the incidence of contaminants in the Cranford samples. Table 2 shows the distribution of the major contaminants in the different size fractions of Italian talc.

TABLE 1. PREVIOUSLY REPORTED⁽¹⁾ PER CENT CONTAMINATION IN TALC SAMPLED AT CRANFORD, NEW JERSEY

Incidence of Contaminants ^(a) , per cent	Date Sampled
< 1	9-6-56, 9-12-56, 9-19-56
1	8-10-56, 9-27-56, 10-18-56, 11-6-56
1-2	10-4-56, 10-29-56
2	8-20-56, 8-28-56, 11-15-56, 12-22-56
2-3	10-12-56, 11-30-56

(a) Determined petrographically.

TABLE 2. THE DISTRIBUTION OF THE MINERAL CONTAMINANTS IN THE DIFFERENT PARTICLE-SIZE FRACTIONS OF ITALIAN TALC

Tyler Mesh Size	Incidence of Contaminants ^(a) , per cent		
	Total	Dolomite	Tremolite
Unseparated	± 2	< 2	Trace
+200	< 1	< 1	0-trace
-200+250	1	1	0-trace
-250+270	1-2	1-2	Trace
-270+325	2	2	Trace
-325+400	2	2	Trace 1
-400	> 2	> 2	< 1

(a) Incidence determined petrographically.

Grit is present in all size fractions, being somewhat more abundant in the fines. In the coarser fractions the mineral contaminants and the talc particles which are oversize in thickness are most readily sensed subjectively. The presence of grit in the fines is largely masked to the senses by the presence of larger platelets. In this regard no solution to abrasiveness lies in the removal of entire coarse size fractions, inasmuch as the grit in the then remaining coarser fractions would be as readily noticeable subjectively and more abundant percentagewise. To remove the abrasive particles, it is necessary to remove both nonplaty talc and the mineral contaminants from the whole powder by such beneficiation methods as flotation⁽⁵⁾ and classification by cycloning⁽⁶⁾, the initial studies on which have been reported or are in preparation (Table 3).

TABLE 3. THE EFFECT ON LUBRICITY OF THE REMOVAL OF MINERAL CONTAMINANTS AND NONPLATY TALC FROM ITALIAN TALC SAMPLES

Talc Sample	Incidence of Indicated Particle Type(a), per cent				Lubricity-Board Measurement, sec
	Platy Talc	Nonplaty Talc	Dolomite	Tremolite	
Italian No. 1					
Feed	88-90	8-9	<2	<1	0.990
Float	95	4	Trace	Trace	1.046
Italian No. 2					
Feed	90	5	3	2	0.926
Float	98	<1	Trace	Trace	1.051

(a) Mineralogical incidence determined petrographically.

It is important to emphasize the difference between the incidence or frequency of contaminants and their per cent of total composition. The per cent incidence is determined petrographically by grain count. It is a two-dimensional measurement approximating area and does not consider the thickness of the particles observed. The incidence of a mineral or crystal type is of primary importance inasmuch as a powder consists of a mixture of discrete grains, each with its particular size, shape, and other physical properties. In considering the behavior of a powder as a lubricant, we are dealing with the mechanical interactions of individual grains in lamellar movement and thus are concerned with the frequency of types of grains, not with their per cent of total composition. That is, for example, in considering lubricity or abrasiveness we must deal with the incidence of individual particles of dolomite rather than with the total volume or weight per cent of the sample which is dolomite, except when dealing with closely sized samples. Conversely, when considering acid solubility, moisture content, and the analysis and evaluation of beneficiation products, we, of necessity, deal with total components; not the incidence of particles. While small differences in per cent incidence of contaminants in a powder may influence the physical properties of mechanical movement, in no case described here is the per cent incidence different from the weight per cent or the chemically analyzed component by more than 1 per cent of the whole sample.

THE MEASUREMENT OF ABRASIVENESS

Discussion

A standard method of measuring the abrasiveness of high-quality talc has not been devised previously. Abrasiveness or grit has been measured subjectively by testing samples between the fingers or teeth. As in the case of lubricity, the final analysis of acceptability in regard to abrasiveness is subjective: consumer reaction. Objective tests are not designed to replace the subjective tests; however, to be able to determine

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improvement in beneficiation procedures and to determine the correlative relationships of the physical properties of talc, it is necessary to be able to measure small differences in the physical properties and to be able to compare them to other quantitative measurements. Knowledge of these interrelationships serves as the basis for interpretation of improvement in quality and thus serves to make it possible to visualize methods of beneficiation.

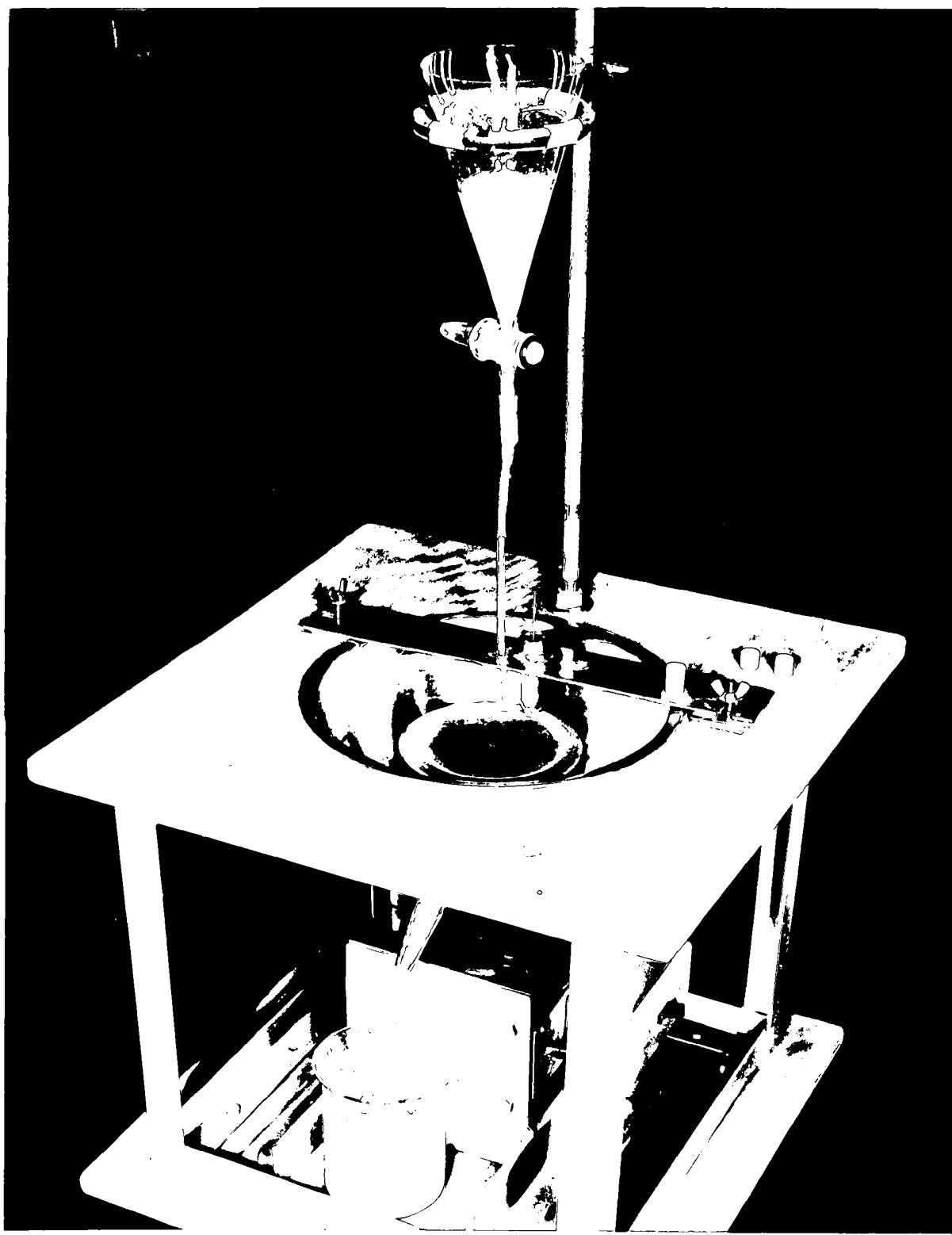
Since the subjective tests are of little help in measuring small differences in one of the many physical properties encountered, and since such tests have no basis for correlation, a machine was built to measure objectively, or test for, abrasiveness, apart from other physical properties.

The Abrasion Machine

Because it was necessary to measure small differences in the abrasiveness of talc, a machine was built to test the wear effect of small concentrations of grit on standard material. The machine was built of a 1/20-hp 1725-rpm electric motor mounted vertically and fitted with a 5-inch lap covered by a Buehler Microcloth held in place by a rubber belt. The lap portion of the machine is set into a steel bowl and covered with a plastic lid. Mounted on a ringstand over the lap a 500-ml open separatory funnel with stopcock is connected by a rubber tube with an adjustable pinch clamp to a feed spout. The separatory funnel contains the sample of talc to be tested in a slurry of 3 grams of talc to 350 ml of water. The feed spout and a cylindrical pellet holder are mounted in a removable crossbar over the lap. Accessibility to these parts is afforded through a hole in the plastic cover. Standard 1/2-inch-diameter pellets are held in the sample holder by a 16.1-gram weight to prevent their skipping or floating on the lap. A 1000-ml beaker mounted under a drain in the steel bowl catches the tested slurry. Figure 1 shows the over-all apparatus. Figure 2 shows the detail of the feed and abrasion mechanism. A detailed description of the abrasion machine and the technique of its operation are found in Appendix A.

In order to measure the abrasiveness of the talc in the slurry a test had to be designed where the object abraded would have a great enough loss to be measured physically. Since the abrasiveness to be measured was that of a powder containing generally from only 1 to 3 per cent of abrasive gangue particles, the material to be abraded had to have a hardness greater than that of the talc, less than that of the grit, and also had to be coherent and homogeneous. After testing a large number of materials it was decided to perform the bulk of the tests on pellets made of minus 400-mesh Italian talc pressed under 50,000-psi pressure. The pellets average 5.20 grams and have dimensions of 1/2 by 7/10 inch. The pellets have a hardness greater than that of the raw talc and less than that of the contaminants (Table 4). Carbonate pellets were made to test specifically for the rarer, harder components, in a similar manner, but using alcohol instead of water in the slurry.

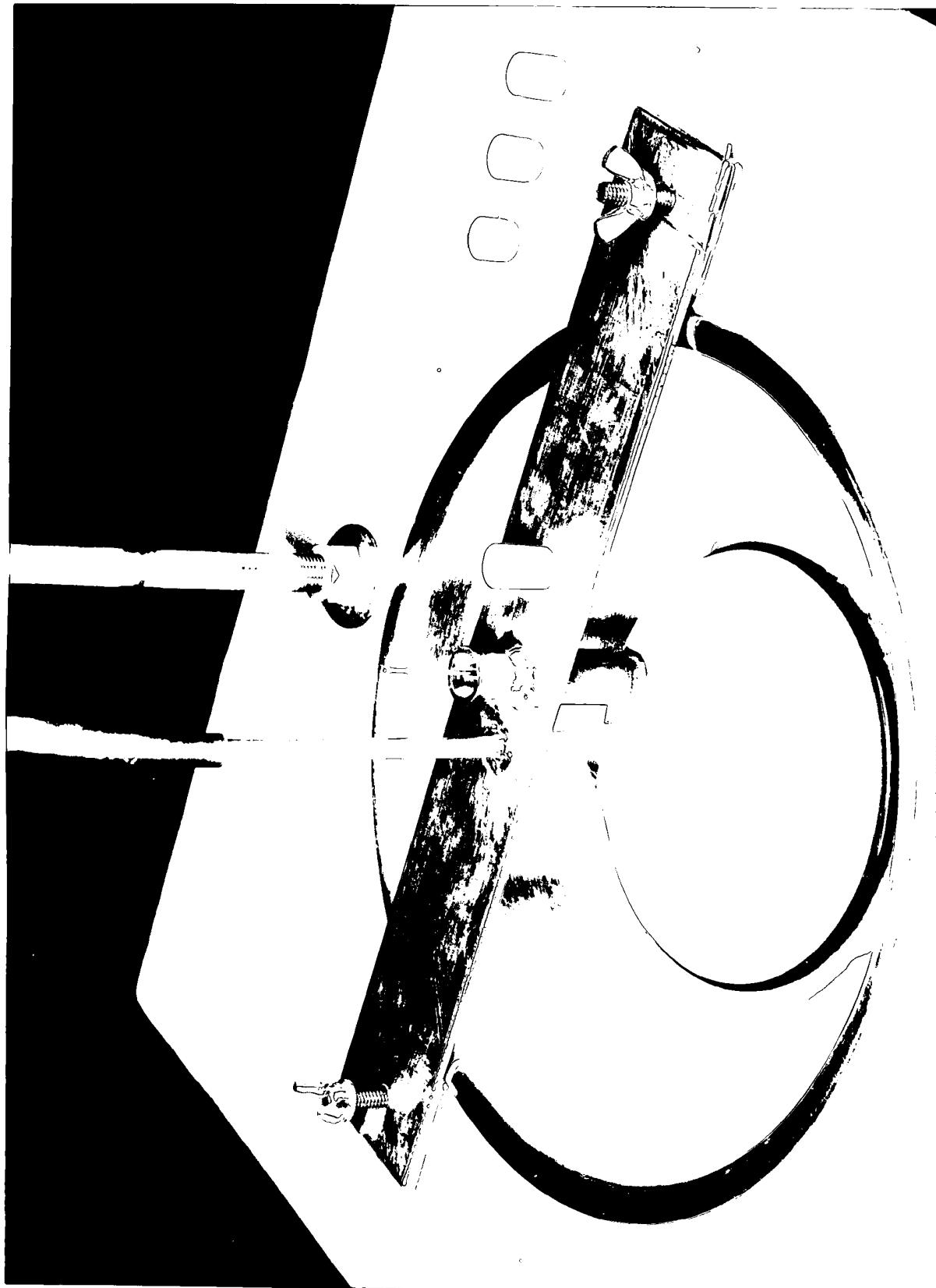
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FIGURE 1. THE ABRASION MACHINE SHOWING RESERVOIR CONTAINING
SAMPLE IN SLURRY TO BE TESTED FOR ITS ABRASIVENESS

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FIGURE 2. DETAIL VIEW OF ABRASION-MACHINE LAP SHOWING FEED SPOUT, CYLINDER IN WHICH PELLETS ARE HELD ON THE LAP, AND STANDARD PELLETS OF PRESSED TALC

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TABLE 4. RELATIVE HARDNESS OF THE TEST PELLETS AND THE GRIT PRESENT IN ITALIAN TALC

Mineral	Moh Hardness
Talc	1
Pressed-talc test pellet	± 2 (scratches talc)
Magnesite	3.5 (scratches talc pellet)
Dolomite	4
Pressed-carbonate test pellet	> 4 (scratches dolomite)
Apatite	5 (scratches carbonate pellet)
Titanite	5
Tremolite	6
Rutile	6
Zircon	7.5

The abrasion-machine operation is timed electrically. The pellets are measured on a micrometer caliper before the test and afterward, after drying. The abrasion measurement is reported in decimal fractions of an inch per second. Although there are limitations to the use of a micrometer, the samples which were compared demonstrated differences in measurement large enough to be significant. Measurements based on weight were found to be entirely unsatisfactory inasmuch as some weight loss was due to spalling and abrasion of the pellet by the walls of the sample tube on portions other than that exposed to the lap and abrasive. This indicated losses which were no indication of the degree of loss due to action on the tested surface alone.

The abrasion machine subjects the standard pellet to abrasion by the sample of talc being studied, at a high rate of speed. It has been calculated that the pellet receives wear equivalent to being rubbed over more than 1800 ft/min of surface of the talc being tested. As expected, the abrasion machine demonstrates that the slurry samples with the greater incidence of mineral contaminants produce the greater amount of abrasion on the pressed pellets. It is also shown that those samples with primarily platy habit are less abrasive than those containing effective amounts of nonplaty talc.

More precise abrasion machines could be built; however, the device used is satisfactory for the purpose of comparing samples within the range of those tested and is an adequate means of obtaining comparable measurements of the effect of grit. Typical figures obtained by the abrasion experiments are shown in Table 5.

TABLE 5. TYPICAL FIGURES OBTAINED BY ABRASION TESTS ON FOUR SAMPLES OF ITALIAN TALC

Test	Incidence of Contaminants(a), per cent	Pellet Measurements, in.		Difference in Measurements, in.	Time, sec	Abrasion(b), 10^{-3} in./sec
		Before	After			
1	2	0.6453	0.5566	0.0887	38	2.33
2	2	0.6435	0.5483	0.0952	41	2.32
3	2	0.6184	0.5374	0.0810	36	2.25
4	2	0.6442	0.5699	0.0743	32	2.32

(a) Determined petrographically.

(b) Significant figure: 2.3×10^{-3} in./sec.The Abrasiveness of Talc Samples

Fifteen samples of talc collected at the Cranford plant of Johnson and Johnson, the same samples used in the previously reported lubricity experiments⁽¹⁾, were tested on the abrasion machine. The results of the measurements are shown in Table 6. The measurements show a range of from 1.62 to 2.69×10^{-3} in./sec wear on the standard pellets. These figures are generally correlative with the incidence of contaminants, as determined petrographically, as reported in the Progress Report dealing with lubricity⁽¹⁾. As it will be shown further in the report, this relationship only holds in whole unseparated powder where particle size is not a fundamental controlling factor. The correlation of lubricity-board measurements with contamination reported in Table 2 of the previous Progress Report⁽¹⁾ shows a similar general relationship between contamination and lubricity. Where the lubricity experiments concluded that the samples containing the greater amount of contaminants demonstrated the poorer lubricity, the abrasion-machine experiments show that the samples with the greater contamination produce the greater amount of abrasion. Thus, when dealing with whole, unscreened powders, the removal of grit should also serve to improve lubricity.

Although the removal of grit improves the lubricity of whole powders, the relationship of lubricity to abrasiveness cannot be considered to be mathematically inverse. The properties which control abrasiveness are the size and shape of the contaminants and their incidence in the coarser fractions; whereas, the properties which control lubricity are the over-all size distribution, those previously described properties directly related to surface area, plus the incidence of the coarser components. When measuring screened fractions of powders both abrasiveness and lubricity are influenced by the specific particle size, and abrasiveness will be directly related to the grit component, whereas in whole powders the finer abrasive particles will be in part masked by the coarser platelets.

To test further the effect of contaminants upon abrasiveness, the contaminants were removed from a sample of talc by froth flotation and the products were tested on the abrasion machine. The same samples had previously been tested for lubricity⁽¹⁾. The test results, which are noticeable subjectively, are reported in Table 7.

TABLE 6. RELATION OF PURITY OF SAMPLE TO ABRASIVENESS AND LUBRICITY IN WHOLE POWDER

Date Sampled	Abrasiveness, 10^{-3} in. / sec	Incidence of Contaminants(a), per cent	Lubricity-Board Measurement, sec
9-12-56	1.62	< 1	1.030
8-10-56	1.70	1	1.021
9-19-56	1.84	< 1	1.028
9-6-56	1.87	< 1	1.083
10-18-56	1.88	1	1.025
9-27-56	1.90	1	1.017
10-4-56	1.90	1-2	0.982
8-20-56	1.91	2	0.971
8-28-56	1.97	2	1.007
11-6-56	2.15	1	1.053
11-15-56	2.30	2	0.936
10-29-56	2.32	1-2	1.006
11-30-56	2.32	2-3	0.952
10-12-56	2.59	2-3	0.968
12-22-56	2.69	2	0.965

(a) Previously reported⁽¹⁾, determined petrographically.

TABLE 7. ABRASION AND LUBRICITY MEASUREMENTS ON FLOTATION PRODUCTS OF ITALIAN NO. 1 TALC

Product	Abrasiveness, 10^{-3} in. / sec	Lubricity-Board Measurement, sec
Starting sample	2.14	0.990
Float product(a)	1.50 (superior)(c)	1.046 (superior)
Nonfloat product(b)	3.03 (inferior)	0.873 (inferior)

(a) Essentially pure talc, representing 90 per cent of starting sample.

(b) 85 per cent talc, 15 per cent contaminants, representing 10 per cent of starting sample.

(c) Less abrasive float products have been made from Italian No. 2 talc.

The float products clearly demonstrate superiority over the starting sample in regard to both abrasiveness and lubricity. The deleterious effect of contaminants is shown by the inferior measurements derived from testing the reject product of the flotation process.

THE RELATIONSHIP OF ABRASIVENESS TO PARTICLE-SIZE DISTRIBUTION AND CONTAMINATION

Discussion

The abrasiveness of the talc studied is determined by its component grit. When dealing with the mechanics of a powder in lamellar motion, we are dealing with the interrelationship of individual particles, and thus are concerned with the per cent incidence and particular sizes and shapes of individuals. The distribution of the contaminants and nonplaty talc in the different size fractions is thus a primary consideration. The coarse contaminants are those which scratch and are quickly noticed subjectively. The finer contaminants clog the lamellar movement of the talc platelets and initiate rolling of the powder, introducing aggregate asperities.

In the previous reports to Johnson and Johnson^(1, 2, 7) the problem of particle-size distribution has been thoroughly discussed. It does not seem requisite here but to re-emphasize the importance of establishing the particle-size distribution of a powder when studying its physical properties or the means of their improvement. Since the size of the abrasive particles, as well as their incidental abundance, contributes to abrasiveness, it was necessary to determine the size distribution of the contaminants.

Correlation of Abrasiveness With Particle-Size Distribution and Contamination

Portions of the same samples which were used to test for lubricity and other physical properties were used in the following experiments on abrasiveness. Size fractions were made of Italian talc and were measured for their abrasiveness on the abrasion machine. Results of the experiments show that the finer particle-size fractions are more abrasive than the coarse. This is in agreement with the incidence of grit determined petrographically. Chemical analyses for equivalent dolomite on these samples are also in agreement. These analyses appear in the section of this report dealing with acid solubility.

Table 8 shows the results of abrasion tests of size fractions on standard talc pellets. When comparing size fractions a parallel relationship exists between lubricity and abrasiveness, as a function of particle size. Thus, were only the fines, the most lubricous fraction, used for baby powder, this fraction would also be the most abrasive. In order to retain the more lubricous particles yet remove the more abrasive, it is necessary to remove the contaminants only. Both lubricity and grittiness cannot be improved by the removal of particle-size fractions.

TABLE 8. RELATIONSHIP OF ABRASIVENESS AND LUBRICITY TO PARTICLE SIZE AND CONTAMINATION IN SIZE FRACTIONS OF ITALIAN NO. 1 TALC

Tyler Mesh Size	Abrasiveness, 10 ⁻³ in./sec	Lubricity-Board Measurement, sec	Incidence of Contaminants(a), per cent		
			Total	Dolomite	Tremolite
Unseparated	2.14	0.990	± 2	< 2	Trace
+200	1.30	0.889	< 1	< 1	0-trace
-200+250	1.59	0.951	1	1	0-trace
-250+270	1.72	0.980	1-2	1-2	Trace
-270+325	2.00	1.030	2	2	Trace
-325+400	2.33	1.043	2	2	Trace-1
-400	2.48	1.099	> 2	> 2	< 1

(a) Determined petrographically.

In order to show which effects are primary, and which are secondarily related because sized materials are analyzed, tests were made on the plus 200-mesh fraction of the talc as received from Italy, on the minus 400-mesh fraction, and on the plus 200-mesh material after it was crushed to pass a 400-mesh screen. The natural plus 200-mesh material had but a trace of contaminants compared with the more than 2 per cent present in the natural minus 400-mesh fraction. Abrasiveness and lubricity tests (Table 9) show clearly that the abrasiveness is controlled primarily by the contamination and only secondarily by the size fraction analyzed. It also shows that the lubricity is controlled primarily by the particle-size fraction tested, and only secondarily by the contamination present in the specific size range. This experiment serves as the basis of interpretation for relating the physical properties of sized material, and also establishes the controlling factors behind lubricity and abrasiveness in whole powders.

TABLE 9. COMPARISON OF LUBRICITY AND ABRASIVENESS TO GRAIN SIZE AND CONTAMINATION

Sample	Abrasiveness, 10 ⁻³ in./sec	Lubricity-Board Measurement, sec	Incidence of Contaminants(a), per cent
Natural +200-mesh fraction	1.30	0.889	Trace
Natural -400-mesh fraction	2.48	1.099	> 2
+200-mesh fraction ground to -400 mesh	1.12(b)	1.108(c)	Trace

(a) Determined petrographically.

(b) The abrasiveness of this sample is only slightly less than that produced by the same sample prior to grinding, much less than the natural minus 400-mesh fraction which contains more grit.

(c) The lubricity of this sample is greatly improved by regrinding, but it is essentially like the natural minus 400-mesh sample despite the difference in the grit present.

TABLE 10. ABRASIVENESS AND LUBRICITY MEASUREMENTS OF ITALIAN TALC SAMPLES FROM WHICH SPECIFIC PARTICLE-SIZE FRACTIONS HAVE BEEN REMOVED

X Represents Fractions Removed From Whole Powder
U Represents Fractions Tested

Tyler Mesh Size	Incidence of Contaminants(a), per cent	Lubricity-Board(b) Measurement of Size Fractions, sec	Abrasiveness, 10 ⁻³ in./sec	Test 1	Test 2	Whole Powder	Test 3	Test 4
+200	<1	0.889	1.30	U	U	U	X	X
-200+250	1	0.951	1.59	U	U	U	X	X
-250+270	1-2	0.980	1.72	U	U	U	X	X
-270+325	2	1.030	2.00	X	U	U	U	U
-325+400	2	1.043	2.33	X	U	U	U	U
-400	>2	1.099	2.48	X	U	U	U	U
Lubricity-Board Measurement, sec								
Abrasiveness, 10 ⁻³ in. / sec								
Approximate Weight Per Cent of Fractions Removed								
97.0 82.0 0.0 2.0 3.0								

(a) Determined petrographically.
(b) Based on Table 10 of previous report(1).

In the previous study of lubricity(1), measurements were made on powders from which different size fractions had been removed, and on mixtures of specific size fractions. The experiment demonstrated that the over-all lubricity was influenced primarily by the coarser particles. Similar experiments on abrasiveness have been made and show that it is not possible to increase the lubricity by removing total size fractions without increasing the abrasiveness (Table 10).

Because talc contains tremolite and rare accessory minerals as well as carbonate as abrasive components, a test was devised to measure the abrasiveness of the harder contaminants. Carbonate pellets were made by fusing a three-to-one mixture of sodium carbonate and sodium borate into a melt. The fusion product was crushed and pressed into pellets under 15,000 psi. The resulting pellets were harder than the talc or the carbonate contaminants but softer than the tremolite and accessory minerals. The pellets were slowly soluble in water; therefore, alcohol was used as the fluid in the test slurries. The test results are not necessarily correlative with measurements made on talc pellets, but demonstrate the distribution of the harder contaminants (Table 11). All other abrasive data contained in this report have been determined on pressed talc pellets.

TABLE 11. THE DISTRIBUTION OF THE CONTAMINANTS HARDER THAN CARBONATE IN ITALIAN TALC AS SHOWN BY ABRASION TESTS MADE ON CARBONATE PELLETS

Tyler Mesh Size	Abrasiveness (on Talc Pellets), 10 ⁻³ in. / sec	Abrasiveness (on Carbonate Pellets), 10 ⁻³ in. / sec	Incidence of Contaminants ^(a) , per cent		
			Total	Dolomite	Tremolite
Unseparated	2.14	0.9	± 2	< 2	Trace
+200	1.30	0.5	< 1	< 1	0-trace
-200+250	1.59	0.7	1	1	0-trace
-250+270	1.72	0.6	1-2	1-2	Trace
-270+325	2.00	0.6	2	2	Trace
-325+400	2.33	0.8	2	2	Trace-1
-400	2.48	0.9	> 2	> 2	< 1

(a) Determined petrographically.

To demonstrate the effect of the rarer contaminants on the abrasion of pressed talc pellets, a series of sized fractions of Italian talc were leached free of the carbonate components and measured on pressed talc pellets. Table 12 shows the degree of abrasiveness produced by the leached samples.

TABLE 12. ABRASION TESTS USING LEACHED AND UNLEACHED SIZED FRACTIONS, DEMONSTRATING THE EFFECT OF DOLOMITE ON ABRASIVENESS

Tyler Mesh Size	Abrasiveness, 10^{-3} in./sec	
	Unleached Powder	Leached Powder
Unseparated	2.14	1.50
+200	1.30	1.34
-200+250	1.59	1.60
-250+270	1.72	1.72
-270+325	2.00	1.71
-325+400	2.33	1.90
-400	2.48	1.75

An additional test, not relative to the beneficiation of Italian talc, but designed as an experiment to serve as a basis for study of lower grade talc, was made on a series of mixtures of Italian talc and minus 400-mesh calcium carbonate (Table 13). Although the data are not to be considered correlative with those of Italian talc, they demonstrate clearly the increasing effect of contamination on abrasion.

TABLE 13. ABRASIVENESS OF MIXTURES OF ITALIAN TALC AND CALCIUM CARBONATE

Per Cent Italian Talc(a)	Per Cent CaO-CO ₂ (-400 Mesh)	Abrasiveness, 10^{-3} in./sec	Difference in Abrasiveness, 10^{-3} in./sec
100	0	2.14	0.75
90	10	2.89	2.20
50	50	5.09	3.08
10	90	8.17	5.48
0	100	13.65	

(a) Contains about 2 per cent native carbonate.

MEASUREMENT AND CORRELATION OF OTHER PHYSICAL PROPERTIES

Moisture Content

The previous Progress Report⁽¹⁾ introduced the problem of moisture content in talc, suggesting that the fine-grain-size fractions should adsorb more moisture on its greater surface area per unit of weight. Table 14 shows the moisture content of size fractions of Italian talc, demonstrating an increase in moisture content with decreasing particle size. Because of the relationship of particle size to other physical properties,

the moisture content of the sized fractions was found to be apparently correlative with a number of other measurements, indicating coincidental relationships which would not hold true in unsized samples.

TABLE 14. RELATIONSHIP OF MOISTURE CONTENT TO PARTICLE SIZE IN ITALIAN TALC

Tyler Mesh Size	Per Cent Moisture(a)
Unseparated	0.05
+200	0.01
-200+250	0.03
-250+270	0.03
-270+325	0.04
-325+400	0.05
-400	0.06

(a) Moisture content determined by method outlined in Johnson and Johnson's Raw Materials Specifications sheet.

The lubricity of talc is related to its moisture content insofar as the moisture content of the finer fractions is higher. In lower grade talc the moisture content was found to be much higher. Six domestic talcs, fabricated to a particle-size distribution similar to that of the Italian talc, showed from 0.08 to 0.20 per cent moisture on analysis. The higher moisture content of some inferior talcs requires that moisture be determined on samples prior to testing for lubricity. Moisture tends to make talc pasty, producing a false indication of superior lubricity on the lubricity board. To demonstrate that the 0.05 per cent moisture content of Italian talc did not affect the lubricity, tests were run on talc from which the moisture had been driven off. A nonreproducible difference of only 0.006 second was recorded, and is not considered to be a significant figure.

To further test the correlation of moisture content and particle size, the talc samples collected at Cranford were analyzed and the data compared with the percentage of fines in the sample. Table 15 compares the moisture content and the percentage of the powder finer than 400 mesh, as compiled from Table 6 of the previous Progress Report⁽¹⁾. The samples with the greater component of fines were found to contain the greater moisture content. No absolute interpretation should be given this relationship. The figures are all very close and their similarity is of greater importance than the correlation. However, there is a theoretical basis for the variance, and the data are presented for whatever they may be worth in the light of future studies. The correlation seems more than coincidental. Moisture content shows no other correlation in whole powders.

Inasmuch as the lubricity-board studies⁽¹⁾ showed that the lubricity variations depended upon the coarser fractions, the small difference in the fine component as related to moisture content should have no expression in lubricity.

TABLE 15. THE RELATIONSHIP OF MOISTURE CONTENT TO PER CENT OF MINUS 400-MESH PARTICLES IN WHOLE SAMPLES OF TALC COLLECTED AT CRANFORD

Per Cent Fines ^(a) (-400 Mesh, Tyler)	Per Cent Moisture	Date Sampled
88.28	0.06	8-28-56
88.16	0.07	9-27-56
87.26	0.06	9-6-56
86.61	0.06	11-6-56
86.09	0.05	9-19-56
85.29	0.05	8-20-56
83.91	0.05	10-18-56
83.40	0.05	12-22-56
83.04	0.05	10-4-56
82.93	0.03	9-12-56
82.57	0.03	11-30-56
82.28	0.04	11-15-56

(a) Repeated from previous report⁽¹⁾.

Johnson and Johnson's Raw Materials Specifications sheet states 0.15 per cent moisture content to be the tolerable upper limit. The Cranford samples, as well as the size fractions, contain considerably less moisture. This indicates that any beneficiation which would change the size distribution, hence the moisture content, would not produce a product of unsatisfactory moisture content.

Inasmuch as a pasty consistency in talcum powder would be undesirable, samples which by exposure or otherwise have taken on excess moisture must be restored by proper drying. One problem arising from flotation experiments was the tendency for the products to agglomerate after drying. It is possible that this moisture can be removed by spray drying. The problem of proper drying is to be considered further in the beneficiation phase of the program.

Absorptive Power

A physical property closely related to moisture content and particle size is the absorptive power of talc. The hygroscopic property is highly important, inasmuch as it is a factor in deodorizing, coloring, in the carrying of perfume or other agents, and in the retention of moisture. Because this subject is only partly understood at this time, it will not be reported on here. The property is not immediately pertinent to the other mechanical and physical relationships in this report except through moisture content.

Alkalinity

The 15 samples of Italian talc collected at Cranford were measured for pH on a Beckman pH meter standardized at neutrality and checked with Beckman buffer solutions of pH 7 and pH 10. The figures are accurate to about 0.1. The samples were prepared by mixing 5 grams of talc with 10 cc of distilled water (pH 6.9). The solutions were agitated and permitted to stand for 2 hours prior to their measurement. The pH of the Cranford samples ranges from 9.0 to 9.3 (Table 16).

TABLE 16. pH OF CRANFORD SAMPLES

Date of Sample	pH
8-10-56	9.1
8-20-56	9.0
8-28-56	9.3
9-6-56	9.1
9-12-56	9.1
9-19-56	9.2
9-27-56	9.2
10-4-56	9.2
10-12-56	9.2
10-18-56	9.0
10-29-56	9.1
11-6-56	9.3
11-15-56	9.0
11-30-56	9.1
12-22-56	9.2

To see if there was any relationship of pH to other physical properties, a Cranford sample with a pH of 9.2 was sized and the fractions were measured (Table 17). The size fractions each measured 9.2, which showed that within the precision of the instrument there was no difference due to particle size or variation in the concentration of carbonates. Studies in progress will determine the practicality of removing the dolomite to the degree that the pH will be lowered. Effective lowering of the pH would lessen Johnson and Johnson's expense of the acid additive.

TABLE 17. pH OF SIZE FRACTIONS OF ITALIAN TALC

Tyler Mesh Size	pH
Unseparated	9.2
+200	9.2
-200+250	9.2
-250+270	9.2
-270+325	9.2
-325+400	9.2
-400	9.2

Acid Solubility

Acid-solubility measurements were made as a part of the study of the carbonate component of Italian talc. It has been demonstrated that carbonate comprises the major amount of the contamination and that its removal decreases abrasiveness and improves lubricity.

The per cent solubility was first determined gravimetrically by the method outlined in Johnson and Johnson's Raw Materials Specifications sheet. The figures obtained were considerably lower than the 6 per cent solubility limit permitted by the specifications; however, they were greater than the figures anticipated from the small amount of carbonate minerals observed petrographically. Solubility analyses determined gravimetrically ranged from 2.10 to 2.81 per cent, showed no relation to any of the physical property measurements. It was assumed that there was either a greater amount of soluble matter in the impalpable fine fraction, or that there was a constant large sample loss during handling.

To resolve the problem the carbonate component was determined petrographically to be primarily dolomite, and a volumetric method of analysis was devised to analyze closely for small concentrations of dolomite in talc (Appendix B). The figures derived from these methods and computations are presented in this report as "equivalent dolomite". It is the measure of the total per cent of dolomite in the sample, not its incidence, which is a function of grain size. The equivalent dolomite analysis is recommended as a substitute for the previously used gravimetric analysis. The method is adjustable for larger concentrations, and other computations may be substituted when carbonates other than dolomite are present.

Equivalent dolomite is correlative with petrographically observed contamination and with related physical properties both in size fractions and in whole powder. A grab sample of Italian talc from the large bulk sample obtained from Cranford, containing slightly higher than average contamination, was analyzed for equivalent dolomite. The analysis showed 1.87 per cent. To check the analyzed percentage against the incidence, a series of grain counts was made on separate immersions, running 1.8, 1.8, 2.0, 1.8, 1.9, and 1.9 per cent. The average 1.8+ is essentially the same as the 1.87 per cent

determined volumetrically. In other cases a concentration of dolomite in the coarse sizes cuts down incidence, as a concentration of dolomite in the fines increases incidence. In the consideration of contaminants in regard to flotation, or of the measurement of lubricity or abrasiveness, the actual incidence of the contaminants is the important consideration. Within the range of Italian No. 1 talc, however, the difference is usually small.

Table 18 shows a comparison of the gravimetric and equivalent dolomite analyses, the incidence of contaminants, and lubricity, as listed against increasing abrasiveness. Table 19 shows contamination and equivalent dolomite compared with decreasing lubricity. These show the primary relationship between abrasion and contamination, and the secondary relationship of abrasiveness to lubricity in whole powders. This was also demonstrated by Table 9 of this report. Table 20 compares the equivalent dolomite, contamination, abrasiveness, and lubricity of sized fractions of Italian talc, demonstrating that the fines contain the more abrasive particles.

Inasmuch as the carbonate in the Italian talc constitutes a rare component in all size fractions, its removal by sizing is not practical. Any practical beneficiation process would be concerned with effectively removing the carbonate from the whole powder, thus improving the slip while eliminating the major abrasive. The effects of flotation on lubricity, abrasiveness, and contamination are presented in a report⁽⁸⁾ on the beneficiation of Italian No. 2 talc.

REFLECTANCE AND WHITENESS

Discussion

The terminology of properties involving the behavior of light is very complex and for the purposes of this report the discussion will be limited to reflectance, whiteness, and gloss. Reflectance is the measurement of the return of light off of a surface in ratio to the intensity of the incident light. This may be measured in terms of brightness, apart from color. Whiteness may be measured in either terms of reflectance over the whole spectrum as "lightness", or in the sense of the absence of specific colors. Gloss is the measure of shininess of surface or specular reflection, as distinct from total reflection.

Gloss, closely related to reflectance and whiteness, will be discussed in a future report when ample samples are prepared to enable assessment of the factors which control the property. Gloss is a separate measurement from those here reported.

Although one may visualize the differences between whiteness, lightness, brightness, and gloss, one cannot subjectively differentiate one from another with any precision or determine the contribution of a specific property to over-all effect. The important consideration is the total subjective effect, which is quickly noticeable. However, in figuring means of improving the over-all effect we must relate the contribution of particle size, shape, and specific contamination to both the over-all effect and to specific properties. For example, fibrous talc is white, but less reflective than platy talc. Rutile is highly reflective, but not white. The beneficiation studies designed to

TABLE 18. COMPARISON OF THE GRAVIMETRIC AND VOLUMETRIC ANALYSES FOR DOLOMITE AND THE RELATIONSHIP OF EQUIVALENT DOLOMITE TO THE INCIDENCE OF CONTAMINANTS AND LUBRICITY, AS LISTED AGAINST INCREASING ABRASIVENESS

Date of Cranford Sample	Abrasiveness, 10 ⁻³ in./sec	Incidence of Contaminants (a), per cent	Equivalent Dolomite (Volumetric), per cent	Acid Solubility (b) (Gravimetric), per cent	Lubricity-Board Measurement (b), sec
9-12-56	1.62	<1	1.5	2.14	1.030
8-10-56	1.70	1	1.5	2.47	1.021
9-19-56(c)	1.84	<1	1.7	2.61	1.028
9-6-56	1.87	<1	1.6	2.64	1.083
10-18-56	1.88	1	1.6	2.44	1.025
9-27-56	1.90	1	1.6	2.39	1.017
10-4-56	1.90	1-2	1.6	2.10	0.982
8-20-56	1.91	2	1.7	2.71	0.971
8-28-56	1.97	2	1.6	2.51	1.007
11-6-56	2.15	1	1.6	2.57	1.053
11-15-56	2.30	2	1.6	2.27	0.936
10-29-56	2.32	1-2	1.6	2.78	1.006
11-30-56	2.32	2-3	1.7	2.81	0.952
10-12-56	2.59	2-3	1.7	2.58	0.968
10-22-56	2.69	2	1.7	2.30	0.965

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- (a) Determined petrographically.
- (b) No relationship demonstrated.
- (c) Petrographically found to contain rare but coarse dolomite.

TABLE 19. EQUIVALENT DOLOMITE, ABRASIVENESS, AND PER CENT CONTAMINATION
LISTED AGAINST DECREASING LUBRICITY

Date of Cranford Sample	Lubricity-Board Measurement, sec	Incidence of Contaminants(a), per cent	Equivalent Dolomite (Volumetric), per cent		Abrasiveness(b), 10 ⁻³ in./sec
			Equivalent Dolomite (Volumetric), per cent	Abrasiveness(b), 10 ⁻³ in./sec	
9-6-56	1.083	<1	1.6	1.87	
11-6-56	1.053	1	1.6	2.15	
9-12-56	1.030	<1	1.5	1.62	
9-19-56(c)	1.028	<1	1.7	1.84	
10-18-56	1.025	1	1.6	1.88	
8-10-56	1.021	1	1.5	1.70	
9-27-56	1.017	1	1.6	1.90	
8-28-56	1.007	2	1.6	1.97	
10-29-56	1.006	1-2	1.6	2.32	
10-4-56	0.982	1-2	1.6	1.90	
8-20-56	0.971	2	1.7	1.91	
10-12-56	0.968	2-3	1.7	2.59	
12-22-56	0.965	2	1.7	2.69	
11-30-56	0.952	2-3	1.7	2.32	
11-15-56	0.936	2	1.6	2.30	

B A T T E L L M E M O R I A L I N S T I T U T E

- (a) Determined petrographically.
- (b) None, or poor correlative relationship.
- (c) Petrographically found to contain rate but coarse dolomite.

TABLE 20. COMPARISON OF CONTAMINATION, EQUIVALENT DOLOMITE, ABRASIVENESS,
AND LUBRICITY IN SIZE FRACTIONS OF ITALIAN TALC

Tyler Mesh Size	Incidence of Contaminants (a), per cent			Equivalent Dolomite (Volumetric), per cent	Abrasiveness, 10 ⁻³ in./sec	Lubricity-Board Measurement, sec
	Total	Dolomite	Tremolite			
Unseparated	±2	<2	Trace	1.9	2.14	0.990
+200	<1	<1	0-trace	0.6	1.30	0.889
-200+250	1	1	0-trace	1.1	1.59	0.951
-250+270	1-2	1-2	Trace	2.0	1.72	0.980
-270+325	2	2	Trace	2.0	2.00	1.030
-325+400	2	2	Trace-1	2.1	2.33	1.043
-400	>2	>2	<1	2.1	2.48	1.099

(a) Determined petrographically.

remove specific particle sizes, shapes, or contaminants, in order to improve the appearance of the talc, will be best controlled when the reflectance and color properties can be assigned to particular components of the powder. As in the lubricity and abrasiveness studies, when the causes of variations are determined, it becomes possible to visualize the means of improving the subjective property.

The reflectance properties of talc begin a new category of measurements. The reflectance properties are distinct from other physical measurements insofar as direct relationships are concerned, except when particle size, surface area, and purity are concerned, as the following experiments demonstrate.

It is apparent at this stage of the investigation that some degree of over-all appearance can be controlled by the selective removal of particular particles.

The Measurement of Reflectance and Whiteness

The Italian talc is nearly pure white and highly reflective. The work under way on reflectance and whiteness is designed to devise a means of improving these properties, particularly in lower grade talc, as the result of interpreting their variations in response to the variations of other physical properties. The program includes determining the effect on whiteness and reflectance of the removal of specific sizes, shapes, and contaminants by beneficiation.

To date the measurements include only those made on a Photovolt Photoelectric Reflection Meter* and on a Gardner Color Meter**. The Photovolt instrument measures diffuse reflectance in terms of "whiteness" or luminous apparent reflectance (LAR). Whiteness in this sense is a matter of lightness without regard to color. A green tristimulus filter is used in the measurement, a standard procedure which permits interlaboratory comparisons. The instrument is calibrated against standard enamel and porcelain plates. The LAR of the Cranford samples is presented in Table 21, showing a range in measurement of 95.0 to 97.5, with no discernible relationship to other properties of the whole powder.

To determine the relationship between LAR and particle size, measurements were made on size fractions, which demonstrated greater reflectance in the fines (Table 22). This indicates that particle size and surface area are important factors. To test if particle shape is also a factor, measurements were made on the products of cyclone classification. These measurements showed that the underflow (platy talc) has a greater reflectance than the overflow (fine acicular talc). A third test made on flotation products demonstrated that purity of sample is also a factor, the float product producing a higher reading than the starting sample. The data related to shape and purity will be included in a report on the beneficiation of talc.

The Gardner Color Meter, among other applications, measures properties designated as Rd and +b. The Rd measurement is one of reflectivity in the sense of brightness, apart from color. The higher the Rd value obtained, the greater the brightness. The +b measurement is one of color based on yellowness, but corresponding to whiteness in near white materials. The lower the +b value the greater is the whiteness.

*Model 610, Photovolt Corporation, New York, New York.

**Gardner Instrument Company, Bethesda, Maryland. This is similar to the instrument used by Johnson and Johnson's Research Laboratory.

Measurements made on the Cranford samples showed a range of 91.30 to 93.25 for Rd and 1.55 to 1.95 for +b, with no correlation as yet established with other physical properties (Table 21).

To test if particle size has any effect on Rd and +b, measurements were made on a series of size fractions, showing that brightness increased in the finer fractions and that whiteness increased with fineness except for the minus 400-mesh fraction (Table 22). In order to interpret the aberrant figure the minus 400-mesh fraction will have to be subdivided and further +b values obtained. It is expected that the concentration of extremely fine acicular particles in the minus 400-mesh fraction accounts for the decrease in the +b measurement. It appears, since 1.60 is the value obtained on the whole powder, that whiteness is lower in the extreme particle sizes, both coarse and fine.

To test the effect of purity of sample on Rd and +b, measurements were made on beneficiated products, showing that the removal of contamination measurably improves Rd and +b. Measurements to be made on cyclone products will demonstrate the effect of particle shape on these properties. These studies will be presented in a forthcoming report on the beneficiation of talc.

Further work is recommended in the matter of improving the sheen of talc. Further investigation is required in the tracing of the specific properties of reflectance to specific particles prior to visualizing beneficiation for the improvement of sheen. It is hoped to be able to adapt a Glossmeter for use on powdered talc in order to be able to correlate properties and plan beneficiation for the improvement of specular reflectance. When sheen can conclusively be traced to specific physical properties of the powder, then beneficiation for its improvement can be visualized.

THE DUST COMPONENT

For the purpose of this report dust may be defined as that fraction of the talcum which remains air borne when the powder is shaken from its container. The dust may be collected for examination by passing a moistened glass slide through the dust cloud which remains suspended in the air when talc is shaken from a container, or by similarly sampling the suspended material after an open container is struck on the bottom onto a table or similar surface. Such action produces two classes of matter, a cloud comprising the bulk of the talc which quickly settles, and a fine portion which does not. Material so collected has been analyzed petrographically and has been found to be composed primarily of platy talc, essentially free of contaminants or acicular particles. This talc represents the finer sizes of platelets - the maximum diameter being about 15 μ in the larger particles. This roughly corresponds to the theoretical $<5-\mu$ sphere fraction⁽²⁾ not including any amount of nonplaty grains or coarser platelets.

The nature of the dust component has been established and it appears likely that it is amenable to beneficiation. Work is at present under way devising a means of comparatively measuring the dust component of talc samples, and to devise a means for its removal should it be practically separable from the whole powder.

TABLE 21. Rd, +b, AND LAR MEASUREMENTS ON THE CRANFORD SAMPLES

Sample Date	Rd	+b	LAR
12-22-56	93.25	1.75	97.5
9-12-56	93.15	1.80	97.0
8-10-56	93.00	1.90	97.0
10-18-56	92.55	1.75	97.5
11-15-56	92.45	1.75	97.0
9-6-56	92.35	1.75	95.0
8-28-56	92.20	1.65	96.0
9-19-56	92.15	1.55	96.0
9-27-56	92.10	1.60	97.0
8-20-56	92.05	1.90	95.5
11-30-56	91.80	1.75	96.0
11-6-56	91.75	1.75	96.0
10-29-56	91.55	1.55	97.5
10-12-56	91.35	1.60	96.0
10-4-56	91.30	1.95	96.0

TABLE 22. Rd, +b, AND LAR AS RELATED TO THE PARTICLE SIZE OF ITALIAN TALC

Tyler Mesh Size	Rd	+b	LAR
Unseparated	91.40	1.60	96.0
+200	85.60	1.90	91.0
-200+250	89.15	1.45	92.0
-250+270	90.36	1.35	92.5
-270+325	90.50	1.30	93.0
-325+400	91.50	1.20	96.0
-400	92.55	1.50	96.0

APPRAISAL OF PHYSICAL-PROPERTY MEASUREMENTS IN THE
EVALUATION OF ORES AND BENEFICIATION PRODUCTS

The foregoing studies, and those previously reported⁽¹⁾, have established the relationships between many of the physical properties of talc and subjective evaluation. Many of the devices employed were helpful in establishing the interrelationships of physical properties, have served their purpose, and their use is not requisite to evaluate the acceptability of talc, inasmuch as the interrelationship of properties permits such an evaluation to be made on the basis of a minimum of measurements.

The subjective tests do not measure specific properties and thus are only of comparative value in deciding what is the specific problem in a nonacceptable talc, or how it may be made acceptable by beneficiation. Such tests, however, must remain the final analysis of acceptability of beneficiation products or in the selection of natural high-grade talcs.

The subjective tests are both a matter of touch and visual comparison. Tested by touch, individual consideration may be given to slip and abrasiveness. Quickly noted in nonacceptable talcs or improper grinds of otherwise acceptable talcs are dry floury feelings, pastiness, the rolling of the powder, poor spread which leaves portions unlubricated, and coarse or sharp grit. Visually it may be quickly noted if the powder is colored or off-white, without sheen, spreads unevenly, contains coarse brilliant particles, or contains a high component of extremely fine dust.

To measure improvement in talc, to maintain quality control, or to visualize proper beneficiation for the improvement of a talc, it is necessary to measure specific physical properties of the powder as a whole, to know the size and shape of the talc particles, and the nature of the contaminants.

Following the subjective appraisal, of foremost importance is petrographic examination. Such a study establishes the platy or nonplaty nature of a talc, identifies the contaminants, and should establish the general size distribution, incidence, degree of subdivision, habit of aggregation, and crystallographic varieties, of the talc, carbonates, amphiboles, and accessory mineral components.

In order to benefitiate for the improvement of slip or the elimination of grit, it is necessary to know the size distribution not only of the crystallographic types of talc present, but also of the different impurities. Size-distribution procedures yield products which may be studied petrographically. These include screening, in the coarser fractions, and sedimentation in the fines. The measurements assigned sedimentation products in usual procedures should be checked petrographically inasmuch as talc platelets behave in the manner of theoretical spheres of much smaller dimensions. A practical method of comparing powders, so long as the theoretical measurements do not become mistaken for actual diameters, is the Andreason sedimentation technique, previously reported⁽²⁾. When this method is employed with supporting petrography it should be a satisfactory device for evaluating beneficiation products.

Without proper size and mineral knowledge of a sample of talc, beneficiation procedures cannot be developed. Control over the physical properties of a talc of known and fairly constant composition could be kept by the use of refinements of the experimental lubricity and abrasion-measuring devices. However, a knowledge of the mineralogy and size distribution is recommended for any talc.

The measurement of surface area, specific surface, porosity, and average diameter will be considered further in regard to compactibility and ullage, and the absorptive power of talc; however, these are not necessary to consider as prerequisite to beneficiation studies for the improvement of the physical properties of talc. The lubricity board and abrasion machine were built to measure small differences in heretofore purely subjective properties and to relate them to established physical measurements. With proper mineralogical and size-distribution knowledge, these properties will be reflected in the other physical measurements.

The following presents the measurements which should be attained in beneficiation products in order to produce material equivalent in quality to Italian No. 1 talc. Improvement of these properties will, of course, produce superior powder, when not improved at the expense of other physical properties. Beneficiation studies on Italian No. 2 talc have produced powder considerably superior to grade No. 1 Italian talc in slip, purity, and the absence of grit.

The following are the recommended requirements for beneficiation products to be the equivalent of Italian No. 1 talc. The items considered important at this stage of the investigation are marked with an asterisk.

Mineralogy*

Platy talc, 90 per cent or more
Nonplaty talc, less than 10 per cent
Carbonates, less than 2 to 3 per cent
Amphiboles, less than 1 per cent
Accessory minerals, trace only
Opaques, none.

Size Distribution*

- (1) Whole powder: Greater than 150 mesh, none
Greater than 200 mesh, less than 1 per cent
Greater than 325 mesh, less than 10 per cent
Greater than 400 mesh, less than 20 per cent.

The powder should have a size-distribution curve over its general range similar to that shown by Andreason sedimentation measurement⁽²⁾ of theoretical particles. Many of the particles finer than the theoretical 5- μ spheres are undesirable, representing fine acicular grains and dust. Fines, however, should not be removed to the extent that the bulk density is raised beyond present specifications.

- (2) Contaminants: Greater than 250 mesh, less than 1 per cent
-250 to +400 mesh, not more than 2 per cent
Finer than 400 mesh, less than 3 per cent.

Note: There is reason to believe that the grind of Italian No. 1 talc is finer than optimum for the production of a superior beneficiated talc. Possibly talc 100 per cent minus 100 mesh would be fine enough. The principal reason for a minus 200-mesh grind for the currently used product may be to reduce the grit to a size where the platelets mask it. With beneficiated talc this would not be necessary and there would be less fines to discard.

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Lubricity Measurement

Greater than 0.93 second, preferably greater than 1 second.

Porosity

Approximately 0.45 to 0.50.

Average Particle Diameter

Approximately 2.4 to 3.3 μ . This measurement is made on the Fisher Subsieve Sizer, the figures are of theoretical particles but are not to be compared with those from the Andreason measurements.

Bulk Density*

22 to 24 lb/cu ft.

Specific Surface, Theoretical

Greater than 6600 cm^2/g , preferable measurements lie in the 8000- cm^2/g range.

Abrasion-Machine Measurement

Talc pellets - less than 2.7×10^{-3} in. / sec, preferably less than 2×10^{-3} in. / sec.

Carbonate pellets - less than 1×10^{-3} in. / sec.

Moisture Content*

Less than 0.08 per cent, preferably 0.05 per cent or less.

pH

Less than 9.4, preferably closer to 7 in order to lower the expense of the acid additive.

Acid Solubility*

Gravimetric, less than 3 per cent
Volumetric, less than 2 per cent.

LAR

95.0 or greater.

Rd*

91.0 or greater.

+b* Less than 2.0. (An additional color measurement, -a, should be taken when talcs with a yellow-green tint are studied.)

The above measurements concern purity, slip, and grit, and the measurement of acceptability of beneficiation products. Yet to be reported on are preferred measurements on the Glossmeter, preferred limits of the dust component, absorptiveness, and compactibility. Although specific problems may arise when other than Italian talc is considered, the above measurements should generally suffice for most raw talcs in the measurement of improvement by beneficiation or of acceptability in regard to Johnson and Johnson's present requirements.

FUTURE WORK

Future work related to the physical properties of talc includes studies of the absorptive power to talc, measurements of gloss as distinct from whiteness and reflectance, measurements of compactibility, the dust component, studies on the effects of different methods of drying processed talc on its physical properties, and further evaluation of the physical properties and mineralogy of beneficiation products.

Because of immediate pressure on other phases of work for Johnson and Johnson most of the above studies will be held in abeyance.

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The original notes on the laboratory work described in this report are in Battelle Laboratory Record Books No. 13034, pages 78 through 96; No. 14187, pages 44 through 100; No. 14431, pages 7 through 100; and No. 14677, pages 1 through 8. The work was done in the period from October 21, 1957, to May 5, 1958.

WLS:djo/gpi/bah

APPENDIX A

DESCRIPTION OF ABRASION MACHINE AND TECHNIQUE OF OPERATION

B A T T E L L E M E M O R I A L I N S T I T U T E

APPENDIX A

DESCRIPTION OF ABRASION MACHINE AND TECHNIQUE OF OPERATION

The experimental device described as the abrasion machine in this report (Figures 1 and 2) consists of a 1/20-hp 220-volt 60-cycle 1725-rpm three-phase Westinghouse electric motor, mounted vertically at operating height, fitted with a steel lap. The steel lap has a diameter of 5 inches and is designed so a lap cloth may be held in place by a rubber belt. A Buehler Microcloth was selected as a standard lap cloth. The lap is housed in a steel bowl 5 inches deep and 9-1/2 inches in diameter. A spout extends from the bottom of the bowl to carry the tested slurry into a beaker. A plastic shield is fitted over the top of the bowl to prevent spatter. A hole in the shield permits observation of the operation and access to the sample holder and slurry feed tube.

The slurry feed tube and a cylindrical 1/2-inch-diameter sample holder are fitted into a metal strip which is fastened in place over the lap. The sample holder is fixed 2 inches from the center of the lap. Directly behind the sample holder the slurry feed tube is fixed in a similar position so that the clockwise rotation of the lap brings the slurry which is to be measured under the standard talc pellet. The talc test pellet is held onto the lap by a 16.1-gram weight which prevents the pellet from skipping or floating on the rotating lap. The slurry feed tube is connected by a rubber tube with an adjustable clamp to a separatory funnel held in a ringstand. The funnel is a 500-ml open-top separatory funnel equipped with a stopcock, and serves as the reservoir for the slurry which is to be measured. The time of operation is kept on a Kodak electric timer.

The pellets are made of minus 400-mesh Italian talc pressed in a F. S. Carver Laboratory Press under 50,000 psi. The pellets are 1/2 inch in diameter, and the 5.2-gram samples used make a pellet about 7/10 inch long.

In addition to the standard talc pellets used in the measurement of total abrasive particles, carbonate pellets were used to measure abrasion by the harder contaminants alone. The carbonate pellets were made by fusing three parts by weight of sodium carbonate to one part of sodium borate into a melt. The fused melt was then crushed and pressed similarly as the talc pellets, under 15,000 psi. Because of swelling during drying the carbonate pellets must be measured wet, unlike the talc pellets. Also, because of slight solubility, the slurry to be tested must be a mixture with alcohol instead of water. The carbonate pellets are less satisfactory than the talc pellets, however, they served a specific experimental purpose.

In operation, the slurry, composed of 3 grams of the talc to be measured, in 350 ml of distilled water, passes onto the rotating lap at a rate controlled by a clamp on the feed tube. The slurry is carried under the standard pellet where the abrasive components wear the pellet at a rate approximating abrasion of the pellet by some 1800 ft/min of surface composed of the sample slurry. Pure samples of talc were found to effect little abrasion, while contaminated talc was found to quickly wear away the pellet. The amount of abrasion loss as measured on a Starrett micrometer caliper is divided by the number of seconds of abrasion to provide figures representing the degree of abrasion.

Should similar experiments be repeated, the following are important considerations. In any series of tests the operation time of the machine should be essentially the same. The talc slurry should be kept in suspension by agitation. A new lap cloth should be used

A-2

as soon as any wear is noticed. All the talc pellets used in a series of tests should be pressed at the same time. Talc pellets should be dried overnight before measuring, to prevent any swelling effects of absorption of water. When any reruns are required on the abrasion machine, the slurry feed should be adjusted to reproduce former readings before comparative data are sought. To make proper comparative measurements the abrasion machine should be operating so as to make replicate tests showing a difference of not more than 0.1×10^{-3} in./sec of abrasion.

APPENDIX B

DETERMINATION OF EQUIVALENT DOLOMITE CONTENT
IN ITALIAN TALC BY VOLUMETRIC ANALYSIS

by

W. E. Brown

BATTELLE MEMORIAL INSTITUTE

Test procedures

5 gram sample - add 0.2 N HCl digest at 120°
for 2 minutes. Heat to boiling. cool. acid must still
remain undiluted.

Filter out solid residue.

Calculation:

(ml HCl x N) = 0.2 N x N. 1 : milliequivalents acid
milliequivalents x 0.0323 = grams HCl used
grams HCl x 1.026 + 1.032 = % carbonates in sample.

5 gram or 1 ml HCl x .92 = % dolomite, etc.
3 " " X 1.54 = " "
5 gram or " X .84 = % magnetite

as Magnetite

grams HCl x 1.15 + 100 = % carbo;

For 5gms milliequivalents x .92 = % dolomite
" " X .84 = % magnetite
" " X 1.00 = % calcite

As magnetite using 2 gram sample.

HCl + 2.1 = % carbonates.

B-1

APPENDIX B

DETERMINATION OF EQUIVALENT DOLOMITE CONTENT
IN ITALIAN TALC BY VOLUMETRIC ANALYSIS

by

W. E. Brown

- (1) Prepare a solution of approximately 0.2N sodium hydroxide and determine exact normality.
- (2) Prepare a solution of approximately 0.2N hydrochloric acid and determine exact normality.
- (3) Weigh out for analysis a 5.000-gram sample of talc and put in a 250-cc beaker.
- (4) Add 25 ml of distilled water to the talc sample and stir with a glass rod to thoroughly wet the talc.
- (5) Add 50 cc of the HCl solution prepared in Step (2). *20 cc*
- (6) Heat sample, containing water and HCl, for 45 minutes at 105 C.
- (7) Raise temperature to boiling for approximately 1/2 minute to expel H_2CO_3 . Use care so that the sample does not boil over.
- (8) Cool to room temperature.
- (9) Add 4 drops of methyl orange indicator to the cooled sample and stir.
- (10) Titrate the sample with the NaOH [from Step (1)] to a yellow end point. This determines the amount of unused acid.
- (11) Calculate the per cent dolomite. An example of the calculations is as follows:

Given: Normality of NaOH = 0.2055 [from Step (1)]
Normality of HCl = 0.2120 [from Step (2)]

Each milliliter of HCl contains $\frac{36.5}{100} \times 0.2120 = 0.0077$ gram of pure HCl

1 ml of NaOH neutralizes 0.97 ml of HCl

1.26 dolomite
1.15 magnesite
1.37 calcite

1 gram of HCl neutralizes 1.26 grams of dolomite

47.6 ml of NaOH was required to titrate a 5-gram sample which had been digested with 50 ml of HCl.

B-2

$47.6 \times 0.97 = 46.17$ ml of unused HCl
 $50.00 - 46.17 = 3.83$ ml HCl consumed by dolomite
 $3.83 \times 0.0077 = 0.0295$ gram HCl consumed by dolomite
 $0.0295 \times 1.26 = 0.0372$ gram dolomite dissolved by HCl
 $\frac{0.0372}{5.000} \times 100 = 0.74$ per cent dolomite.

Note: In order to test the accuracy of this method of analysis, some relatively pure dolomite (taken from a mineral specimen) was analyzed. The weight of the sample analyzed was 0.0300 gram. The foregoing analytical method showed the sample to contain 0.0306 gram of dolomite. Another check test was made by analyzing a sample of Italian talc for per cent of CO₂, and converting the CO₂ content to the theoretical amount in dolomite. The CO₂ analysis indicated that the dolomite content was 2.26 per cent. By volumetric analysis the dolomite content was calculated to be 2.18 per cent.

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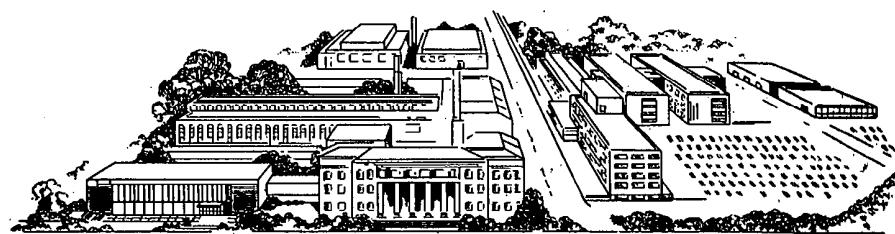
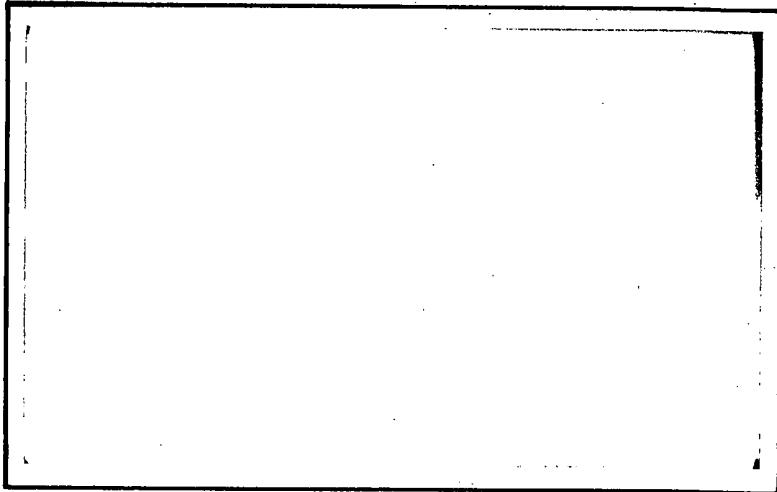
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Exhibit 30

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PHASE REPORT

on

**PILOT-PLANT BENEFICIATION OF ITALIAN
RUN-OF-MINE TALC**

to

JOHNSON AND JOHNSON

March 8, 1960

by

R. W. Schatz

**BATTTELLE MEMORIAL INSTITUTE
505 King Avenue
Columbus 1, Ohio**

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Battelle Memorial Institute

505 KING AVENUE COLUMBUS I., OHIO

March 28, 1960

Mr. W. H. Ashton
Johnson and Johnson
Research Department
New Brunswick, New Jersey

Dear Mr. Ashton:

We are pleased to transmit six copies of our Phase Report, "Pilot-Plant Beneficiation of Italian Run-of-Mine Talc".

As pointed out in the report, the runs were quite successful in that a superior talc product of high luster and plateness was obtained at a high recovery.

Sincerely yours,



O. F. Tangel

OFT/mln
Enc. (6)

cc: Dr. W. H. Lycan
Mr. C. V. Swank

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TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION AND SUMMARY	1
SCOPE OF PILOT-PLANT WORK.	2
GRINDING INVESTIGATION	2
Crushing	2
Grinding	2
BENEFICIATION	7
OPERATIONAL PROBLEMS	7
CHARACTERISTICS OF PILOT-PLANT PRODUCTS.	14
SAMPLES SHIPPED TO JOHNSON AND JOHNSON	14
CONCLUSIONS AND FUTURE WORK.	14

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PHASE REPORT

on

PILOT-PLANT BENEFICIATION OF ITALIAN
RUN-OF-MINE TALC

to

JOHNSON AND JOHNSON

from

BATTELLE MEMORIAL INSTITUTE

by

R. W. Schatz

March 8, 1960

INTRODUCTION AND SUMMARY

Laboratory work* had shown that a superior talc product could be produced by wet grinding and flotation of Italian run-of-mine** talc. Consequently, Italian run-of-mine talc was processed in the pilot plant to demonstrate the technical feasibility of the process and to obtain drum quantities for panel testing.

The treatment procedure was as follows: after hammermill crushing to minus 1/4 inch, the talc was ground wet in a pebble mill, in closed circuit with cyclones, to approximately 98 per cent minus 200 mesh. After removal of the major portion of the minus 10-micron fraction in other cyclones, the platy talc was recovered by flotation, filtered, and spray dried. Sixty to sixty-five per cent by weight of the feed to the plant was recovered as a superior talc product. Deionized water was used throughout the process.

The beneficiated talc was at least 99 per cent platy, analyzed from 0.2 to 0.3 per cent CO₂, contained about 12 to 13 per cent of minus 10-micron material, and had a luster higher than either the presently sold baby powder or the beneficiated Italian No. 2 talc.

* Progress Report, "The Physical Concentration of Talc Ores-Flotation of Italian Run-of-Mine Talc", December 31, 1959.
** Hereafter abbreviated as ROM.

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2

SCOPE OF THE PILOT-PLANT WORK

The pilot-plant study of the beneficiation of Italian ROM talc was composed essentially of two phases. The first was to determine the operating conditions required in the grinding circuit to produce a suitable wet-ground talc for flotation. The second was to establish flotation conditions for the beneficiation of the wet-ground talc. The first phase was completed in the last half of September and the first half of October, 1959. The flotation phase was completed by the middle of November, 1959.

GRINDING INVESTIGATION

The grind desired on the Italian ROM talc was 98 per cent minus 200 mesh with a minimum of minus 10-micron material. Equipment in the crushing and grinding circuit included a hammermill (4)*, pebble mill (8), two cyclones (10, 10a) together with the necessary pumps (9, 9a), conveying (3-5), storage (6), and feeding (7) facilities. A feed rate of about 450 pounds per hour to the grinding circuit was desired because this rate was the approximate capacity of the beneficiation section of the pilot plant.

Crushing

The ROM talc was received in burlap sacks (150 pounds per sack) and had been crushed to about minus 2 inches. In order to maintain a fixed storage bin level, three sacks of talc were crushed per hour; two on the hour, one on the half hour. The hammermill product was nominally minus 1/4 inch in size. Table 1 presents typical screen analyses of the ROM talc before and after hammermill crushing.

Grinding

The following general plan was used to determine the operating conditions required for the desired grind. For each set of conditions the circuit was first operated for about 4 hours to establish equilibrium. After equilibrium had been reached, as determined by pulp density and volume measurements throughout the circuit, the required samples were taken over a period of 2 to 3 hours. Based on the analyses of these samples, changes were made in the operating conditions, and after equilibrium was reached, the circuit was again sampled. This procedure was repeated until the desired operating conditions were established.

* For equipment specifications see corresponding identification number in the Phase Report "Design and Construction of a Talc Flotation Pilot Plant", December 30, 1959.

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3

The initial tests, started on September 18, 1959, were made using the pebble mill (8) with 1500 pounds of 1-1/4-inch porcelain balls and the 4-inch (No. 1) short cyclone (10). It was soon apparent that insufficient grinding was being obtained in the mill, and, therefore, the pebble load was increased to 2900 pounds. Additional runs were made using the one cyclone with various apex (discharge) openings, feed and overflow discharge pressures, and pulp densities. The finest grind reached was 90 per cent minus 200 mesh with about a 20 per cent minus 10 microns.

It appeared that, to reach the desired grind, it would be necessary to use No. 1 and No. 2 cyclones in series, i. e., the second cyclone retreating the overflow from the first, with the underflow from both cyclones returning to the pebble mill. After considerable experimentation with this circuit, operating conditions were established which gave a grind of 98 per cent minus 200 mesh with 25 to 30 per cent minus 10 microns. This grinding investigation was completed on October 15, 1959.

Figure 1 shows the flowsheet of the grinding circuit. Typical operating data, as well as an approximate water-solids balance also are shown.

Table 1 gives typical screen size analyses for the various points in the grinding circuit. Tables 2 and 3 present additional operating data: volumes, pump densities, water requirements, cyclone operating pressures, and efficiency calculations.

The circulating load and classification efficiencies of Table 3 are worthy of comment. The circulating load of 660 per cent is rather high for a conventional ball mill-cyclone circuit. In addition, the classification efficiency of 21 per cent (based on recovery of minus 200-mesh material fed to the cyclone circuit) is low.

At least two factors contribute to these conditions. One is the platy character of talc. As particle shape deviates from a sphere or cube, classification becomes more difficult, and in a one-pass operation more inefficient. In addition, a 98 per cent minus 200-mesh grind with less than 0.3 per cent on 100 mesh is a difficult grind to reach, particularly with a platy material. Excellent rejection of the plus 200-mesh fraction must be obtained, and this is only accomplished at the expense of recirculating a considerable quantity of finished (minus 200 mesh) product to the pebble mill. The efficiency of rejection of plus 200-mesh material was excellent: 99 per cent, as shown in Table 3.

Further experimentation with the grinding circuit might lead to a grind of 98 per cent minus 200 mesh with less than 25 to 30 per cent of 10-micron material, as well as lower circulating loads and improved efficiencies. For an investigation of this sort, 24-hour (continuous) operation would be necessary with minor changes in the circuit from time to time. This type of an investigation is one that is best made in an operating plant because the effect of each minor change will be small and a number of days of operation under each set of conditions are necessary to establish the effect of the change. It is Battelle's opinion that improvements in the grind can be made which will reduce the quantity of minus 10-micron talc.

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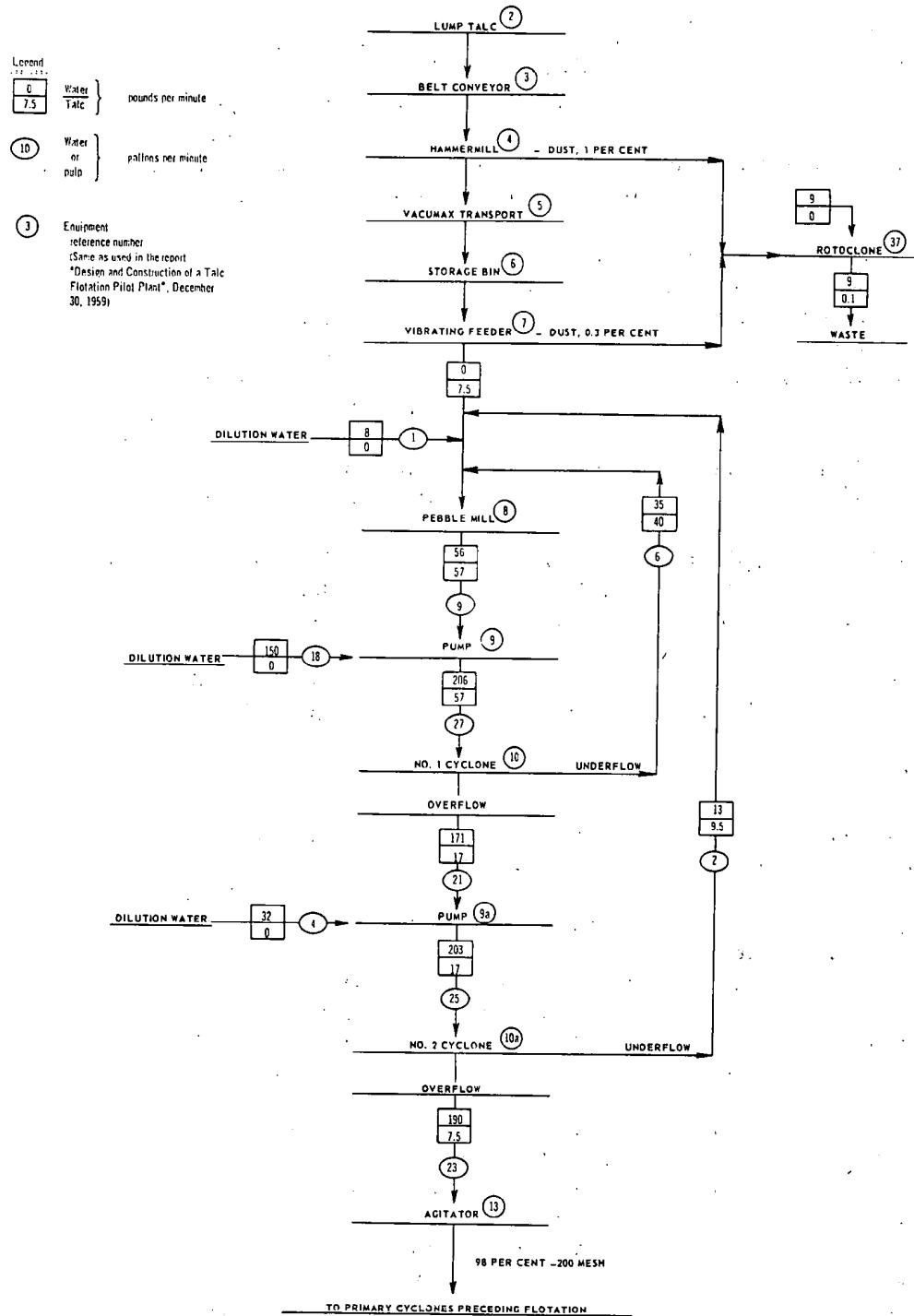


FIGURE 1. PILOT-PLANT GRINDING CIRCUIT FLOWSHEET FOR TREATMENT OF ITALIAN RUN-OF-MINE TALC

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TABLE 1. TYPICAL SIZE-DISTRIBUTION DATA, CRUSHING AND GRINDING CIRCUIT^(a)

Pilot-Plant Treatment of Italian Run-of-Mine Talc

Mesh	Size Distribution, per cent by weight						
	Hammermill		Ball-Mill Discharge	No. 1 Cyclone		No. 2 Cyclone	
	Feed ^(b)	Product ^(c)		U'flow	O'flow	U'flow	O'flow ^(d)
+ 3	41.8	--	--	--	--	--	--
- 3+ 10	22.7	12.0	tr	tr	--	--	--
- 10+ 20	11.7	15.3	0.4	0.4	--	tr	--
- 20+ 35	5.8	17.3	1.7	2.5	--	tr	--
- 35+ 65	6.6	16.8	8.3	10.9	5.0	0.5	tr
- 65+100	2.9	6.5	14.5	16.3	3.2	3.2	tr
-100+200	3.2	6.1	14.2	14.5	2.2	10.4	2.0
-200	5.3	26.0	60.9	55.4	89.6	85.9	98.0
- 10 microns	0.3	1.0	6.8	--	--	--	29.6

(a) Run of October 22, 1959.

(b) All passing 2 inch.

(c) Ball-mill feed.

(d) Feed to primary cyclones preceding flotation.

TABLE 2. TYPICAL GRINDING-CIRCUIT OPERATING DATA

Pilot-Plant Treatment of Italian Run-of-Mine Talc

Circuit	Solids, lb/min	Weights, Volumes, and Pulp Densities		New Water, gpm
		Slurry Solids, %	GPM	
Pebble Mill				
New Feed(dry)	7.5	--		1
Discharge	--	51	9	--
No. 1 Cyclone				
Feed	57	22	27	18
Overflow	17	9	21	--
Underflow	40	53	6	--
No. 2 Cyclone				
Feed	17	8	25	4
Overflow	7.5	4	23	--
Underflow	9.5	42	2	--

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6

TABLE 3. TYPICAL GRINDING-CIRCUIT OPERATING DATA

Pilot-Plant Treatment of Italian Run-of-Mine Talc

Grinding-Circuit Circulating Load

$$\text{Per cent circulating load} = \frac{\text{Weight cyclone underflow}}{\text{Weight new feed}} \times 100$$

New feed = 7.5 lb/min

Underflow, No. 1 cyclone = 40 lb/min

Underflow, No. 2 cyclone = 9.5 lb/min

$$\text{Per cent circulating load} = \frac{40 + 9.5}{7.5} \times 100 = 660\%$$

Grinding-Circuit Classification Efficiency
(At 200 Mesh)

Recovery of Minus 200-Mesh Fraction

$$\text{Efficiency, \%} = \frac{\text{Weight of minus 200-mesh No. 2 cyclone overflow}}{\text{Weight of minus 200-mesh No. 1 cyclone feed}} \times 100$$

$$\text{Efficiency, \%} = \frac{7.5 \times 0.98}{0.57 \times 0.609} \times 100 = 21.2\%$$

Rejection of Plus 200-Mesh Fraction

Efficiency, \% =

$$\frac{(\text{Weight of plus 200-mesh No. 1 cyclone feed}) - (\text{Weight of plus 200-mesh No. 2 cyclone overflow})}{\text{Weight of plus 200-mesh No. 1 cyclone feed}}$$

$\times 100$

$$\text{Efficiency, \%} = \frac{(57 \times 0.391) - (7.5 \times 0.02)}{57 \times 0.391} \times 100 = 99.3\%$$

Cyclone Operating Data

	Gpm Feed	Feed	Overflow	Underflow
No. 1 Cyclone(a)	27	22	2	(c)
No. 2 Cyclone(b)	25	16	6	(c)

(a) 4-inch short cyclone - 9/16-inch inlet

1-inch vortex finder

3/4-inch apex.

(b) 4-inch long cyclone - 3/4-inch inlet

3/4-inch vortex finder

3/8-inch apex.

(c) Free discharge.

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BENEFICIATION

Beneficiation of the wet-ground ROM talc was started on October 19, 1959. The first circuit used was that employed in the concentration of Italian No. 2 talc.*

Microscopic examination of the froth from each cell showed that the talc being floated in all of the first 6 flotation cells was of good quality and that recirculation of the concentrate from Cells 5 and 6 (as practiced on Italian No. 2) was not necessary. Consequently, circuit changes were made which eliminated Float 2, the scavenger cyclones, and the scavenger flotation cells. Thus all 6 cells of the previous Float 1 and Float 2 circuits became Float 1.

After several hours of operation with this straight rougher (Float 1) circuit on October 21, it was apparent that insufficient quantities of flotation reagents were being used. (This was because there was now no circulating load to return reagents to the flotation circuit.) Additional amounts of reagents were added for the balance of the shift. The low weight recovery, 49 per cent, obtained on this day is believed to be the result of several hours of operation with insufficient reagents.

This circuit, as shown in Figure 2, was operated on October 22, November 3, and November 4 with no changes. On November 5 and 6 reagents were increased 10 per cent. The metallurgical results obtained during these 6 days of operation are given in Table 4. Recoveries of 60 to 65 per cent, based on plant feed, were obtained in producing a superior talc product.

Typical operating data are given in Figure 2 and Tables 5, 6, and 7. These include size analyses, weights, volumes, pulp densities, cyclone operating data, primary cyclone efficiency calculations, and flotation reagent quantities.

OPERATIONAL PROBLEMS

The usual start-up and break-in problems were encountered in the grinding circuit, but once these had been solved, the entire circuit ran smoothly. Minor problems that occurred, and should be kept in mind in designing a commercial plant, have been discussed in the Phase Report of December 30, 1959, "Design and Construction of a Talc Flotation Pilot Plant".

* Phase Report, "Pilot-Plant Beneficiation of Italian No. 2 Talc", March 1, 1960.

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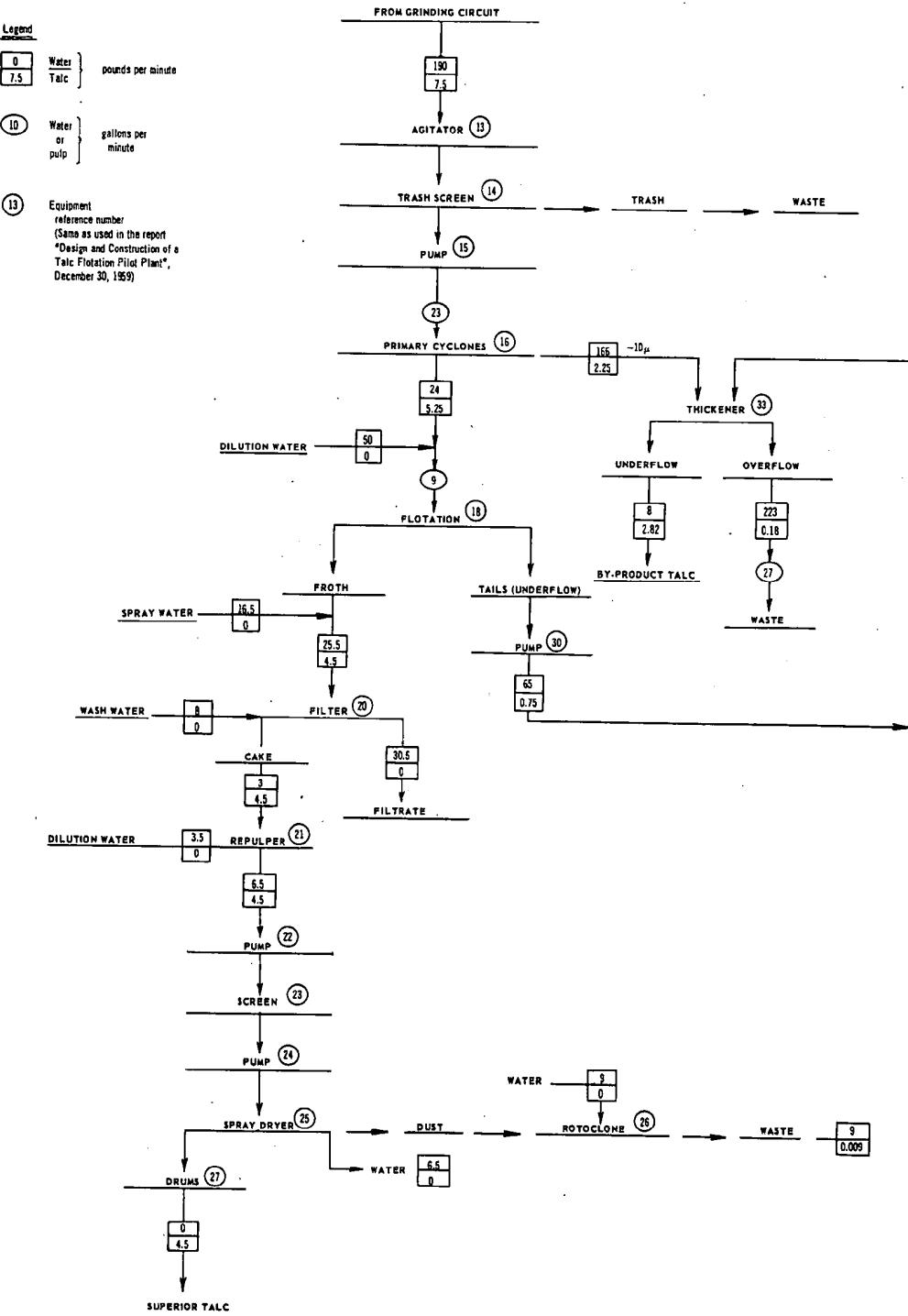


FIGURE 2. PILOT-PLANT FLOTATION-CIRCUIT FLOWSHEET FOR TREATMENT OF ITALIAN RUN-OF-MINE TALC

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9

**TABLE 4. SUMMARY OF PILOT-PLANT BENEFICIATION OF ITALIAN
RUN-OF-MINE TALC**

Date, 1959	Oct. 21	Oct. 22	Nov. 3	Nov. 4	Nov. 5	Nov. 6
Feed Rate, lb/hr	438	432	467	440	442	468
Operating Time, min	359	380	366	390	390	330
Feed(a)						
Platy Talc, %	91-92	91-92	91-92	91-92	91-92	91-92
CO ₂ , %	0.61	0.64	0.68	0.61	0.68	0.61
-10 Micron, %	29.5	29.6	27.0	25.2	28.9	26.2
Beneficiated Talc						
Platy Talc, %	99	99	99	99	99	99
CO ₂ , %	0.15	0.17	0.26	0.19	0.32	0.28
-10 Micron, %	9.0	10.4	13.0	13.6	13.0	12.4
Over-All Weight Recovery, % ^(b)	48.8	58.0	61.8	61.1	61.3	65.9
Luster	nd	nd	1.59	1.56	1.54	1.59
Combined Tailings(c)						
CO ₂ , %	1.05	1.29	1.36	1.27	1.25	1.25
-10 Micron, %	61.9	59.0	nd	nd	nd	nd

(a) Grinding-circuit cyclone overflow.

(b) Based on CO₂ analyses.

(c) Primary cyclone combined with flotation tailing.

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10

TABLE 5. TYPICAL SIZE-DISTRIBUTION DATA, FLOTATION CIRCUIT^(a)

Pilot-Plant Treatment of Italian Run-of-Mine Talc

Mesh	Primary Cyclones		Flotation Tailing	Combined Tailings ^(c)	Dryer Product
	U'flow ^(b)	O'flow			
+ 65	1.0	--	--	--	0.2
- 65+100	1.6	--	--	--	1.3
-100+200	0.6	--	--	--	1.0
-200	96.8	--	97.9	98.7	97.5
- 10 microns	9.4	71.2	15.4	59.0	10.4

(a) Run of October 22, 1959.

(b) Flotation feed.

(c) Flotation tailing combined with primary cyclone overflow.

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11

TABLE 6. TYPICAL PRIMARY-CYCLONE AND FLOTATION-CIRCUIT OPERATING DATA

Pilot-Plant Treatment of Italian Run-of-Mine Talc

Circuit	Weights, Volumes, and Pulp Densities			New Water, gpm
	Solids, lb/min	Slurry Solids, %	GPM	
Primary Cyclones				
Feed	7.5	4	23	--
Overflow	2.25	1.4	20	--
Underflow(a)	5.25	7	9	6
Flotation				
Feed	5.25	7	9	--
Froth(b)	4.5	15	3	2
Tailing	0.75	1.3	8	--
Filtration				
Feed	4.5	15	3	1(c)
Filtrate	0	0	3.6	--
Cake	4.5	60	0.4	--
By-Product Thickener				
Feed	3.0	1.3	28	--
Overflow	0.18	--	27	--
Underflow	2.82	26	1	--

Primary Cyclone Classification Efficiency
(At 10 microns)

Rejection of Minus 10-Micron Fraction

$$\text{Efficiency, \%} = \frac{\text{Weight of minus 10 microns in cyclone overflow}}{\text{Weight of minus 10 microns in cyclone feed}} \times 100$$

$$\text{Efficiency, \%} = \frac{2.25 \times 0.712}{7.5 \times .296} \times 100 = 72.1\%$$

Cyclone Operating Data

	Pressure, psig			
	GPM	Feed	Overflow	
Primary Cyclones	23	32	10.5	6.5

(a) After dilution water.

(b) After launder spray water.

(c) Wash water on filter.

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12

TABLE 7. TYPICAL FLOTATION-REAGENT DATA

Pilot-Plant Treatment of Italian Run-of-Mine Talc

Reagent	Conditioner	Flotation Reagents, pounds per ton new feed						By-Product Thickener	Total		
		Flotation Cells									
		1	2	3	4	5	6				
HCl, 37 % ^(a)	±1.0 ^(e)								±1.0 ^(e)		
Dowfroth 250 ^(b)	0.06	--	--	0.06	0.06	0.03	--		0.21		
Aerosol 18 ^(c)	0.24	--	0.24	--	0.12	--	--		0.60		
Separan AP30 ^(d)	--	--	--	--	--	--	--	0.10	0.10		

(a) 10 % solution.

(b) 0.5 % solution.

(c) 2 % solution.

(d) 0.2 % solution.

(e) To pH 6.9-7.1.

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13

TABLE 8. CHARACTERISTICS OF TALC PRODUCTS

	Code	Petrographic Analysis, per cent(a)					Wt % $\text{-}10\mu$ Luster
		Platy Talc	Nonplaty Talc	Carbonate	Tremolite	CO_2 , % (b)	
ROM talc, untreated	--	91-92	5	1-2	1	0.6-0.7	
Beneficiated ROM talc	ST94-95	>99	<1	<1	tr	0.22-0.24	11.3 157-153
Primary cyclone overflow	BP4	25	77	2-3	± 1	0.70	70.0 nd
Flotation tailing	BP5	83-85	6-8	7-9	1-2	2.68	14.0 nd
Combined by-products(c)	BP6	73	23	2-3	1-2	1.44	55.2 nd
Johnson and Johnson shelf product(d)		89	9	<2	tr	0.55	25-30 1.34
Italian No. 2 beneficiated	ST27-28	99	<1	<1	tr	0.33	8.7 1.47

(a) Determined by mineral count.

(b) Chemical analysis.

(c) Primary cyclone overflow combined with flotation tailing.

(d) Sample of August, 1958.

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14

CHARACTERISTICS OF PILOT-PLANT PRODUCTS

Table 8 lists the characteristics of the various talc products. The beneficiated talc was made on November 6 while the by-products were obtained on November 3 and 4. For comparison purposes, similar data are presented for the beneficiated talc from Italian No. 2 and the present Johnson and Johnson shelf product.

The outstanding property of the superior talc from ROM as compared with the Italian No. 2 concentrate is the appearance of the ROM talc. It is flakier and of higher luster. The higher luster apparent by eye also is confirmed by the luster measurements. The average luster for the superior talc over the period November 3 to 6 on ROM was 1.57; whereas for Italian No. 2 for the period August 13 to 21, it was 1.47.

SAMPLES SHIPPED TO JOHNSON AND JOHNSON

Approximately 360 pounds of superior talc produced on November 6 and three 15-pound samples of by-products (primary cyclone overflow, flotation tailings, and combined tailings) obtained on November 3 and 4 were shipped to Johnson and Johnson on November 25 and 27. Data on these samples are given in Table 8.

CONCLUSIONS AND FUTURE WORK

The pilot-plant beneficiation of ROM talc was completely successful in that a superior product representing about 60 to 65 per cent of the feed was obtained with little difficulty. Improvement in quality of product (particularly in regard to lowering the 10-micron content) probably cannot be expected by altering the flotation conditions without a sacrifice in recovery. On the other hand, increased recovery might be obtained by recycloning the primary cyclone overflow to recover some of the platy talc that it contains. Improvements in the grinding circuit to lessen production of 10-micron material would also increase recovery. Either or both of these improvements should not lower the quality of the beneficiated talc. From an operational cost standpoint, use of raw water or re-use of process water would appear to be the best areas for future investigations.

The original data for this report are to be found in Notebook No. 16330, pages 1 to 45. The work period covered is from September 14 through December 1, 1959.

RWS/mln

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JNJ000086877

Metadata

AttachCount	0	ORIGINAL
BegAttach	JNJ 000086877	ORIGINAL
Confidentiality	N	ORIGINAL
Custodian	Bill Ashton c/o Lorena Telofski	ORIGINAL
DateMod	03/08/1960 12:00 AM	ORIGINAL
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PgCount	20	ORIGINAL

Exhibit 31

Battelle Memorial Institute

5 0 5 K I N G A V E N U E C O L U M B U S , I., O H I O

June 6, 1961

Mr. W. H. Ashton
Research Department
Johnson and Johnson
New Brunswick, New Jersey

Dear Mr. Ashton:

This letter briefly summarizes my observations made during May at the Wight Mine, Gouverneur, N. Y.; the Hammondsville, Vt., deposit; and subsequent petrographic examinations of the ores and beneficiation products. The following covers only such information as I believe is of immediate consequence to you. A detailed geological and mineralogical report will follow if you so request.

THE GOUVERNEUR, N. Y., DEPOSIT

Talc deposits in the Gouverneur belt constitute the largest in the Western Hemisphere. In most of the Gouverneur deposits talc is subordinate. The ore is tremolite-anthophyllite schist which has been serpentized and steatized. The ore contains tremolite, anthophyllite, talc, serpentine, hexagonite, quartz, carbonates, micas, pyroxenes, and opaques. The talc belt is some six miles long, extends down dip over 2000 feet, and has widths over 400 feet. The particular talc of interest to Johnson & Johnson is a tremolitic platy talc in the Wight Mine. This facies is objectionable in the International Talc Company product and is avoided in mining. The body is 12 to 14 feet thick, extends from near surface through the 6th level and is presumed continuous along the strike of the ore. Although no attempt has been made to block out the platy ore, I was able to see a probable 100,000 tons, 300,000 tons can be estimated from drilling data and intersections of the present workings. Based on information provided by the International Talc Co., the reserves may be in excess of 1/2 million tons. The tonnage figures are only rough estimates. The ore reserves and the uniformity of the ore would have to be proven by drilling.

Flotation Concentrate from Gouverneur Talc

The ore contains both tremolite and platy talc as major components. The flotation concentrates you submitted contained:

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Battelle Memorial Institute

Mr. Ashton

-2-

June 6, 1961

Total talc	96-97% (by count)
Platy talc	93
Nonplaty talc	3
Free tremolite, others	3-4

However, the talc platelets contain abundant fine tremolite which is liberated on successive fine grinding. Although the beneficiated talc is of high quality, it would appear that 4 or 5 per cent tremolite is the minimum which would be practically obtainable without extensive changes in the proposed beneficiation procedure.

THE HAMMONDSVILLE, VT., DEPOSIT

The ore previously mined at the Hammondsdale deposit was off-color, highly contaminated, and unsuitable for producing high-grade talcum. When the old talc body was exhausted, mining proceeded underground and struck a white platy talc, so far unique in Vermont. The character of the new ore was reportedly not recognized until some two months ago. The ore is enclosed in quartz-mica schist, consists of some 70-75 per cent platy talc with inclusions of coarse carbonate grit (about 25 per cent) and accessory minerals.

The white, platy ore has not been blocked out, however, about 100,000 tons can be visualized from the present workings. If the talc body is as large as visualized by the Eastern Magnesia Talc Co., there may be as much as 1/2 million tons or more. The tonnage figures are only rough estimates. The ore reserves and the uniformity of the ore would have to be proven by drilling.

Flotation Concentrate from the Hammondsdale Ore

Samples of the Hammondsdale ore were processed by flotation by the Eastern Magnesia Talc Co. The flotation concentrate contained:

Total talc	97-98% (by count)
Platy talc	96
Nonplaty talc	<2
Carbonates	<1
Rutile (liberated)	<1
Altered amphiboles	trace
Opaques	trace
Others	<1

Examination of several specimens from different parts of the mine shows most of the ore to be free of rutile. Some specimens show 4-5 per cent rutile. On insufficient evidence the rutile appears to be traceable, for the most part, to a specific stope on the footwall.

Battelle Memorial Institute

Mr. Ashton

-3-

June 6, 1961

EVALUATION

There are now two known domestic sources of talc ore which may be beneficiated to high-grade talcum and which appear to have adequate reserves. The flotation concentrates produced from the platy Gouverneur ore and the new Hammondsville ore have very good slip, color, and are primarily composed of single crystalline sheets.

Before a final conclusion is reached, the following should be considered:

- (1) The reserves of either mine should be blocked out and core drilled to establish uniformity.
- (2) It should be established whether or not the tremolite content of the Gouverneur concentrate, if prohibitive, can be removed economically by further beneficiation.
- (3) It should be established how the rutile is distributed throughout the Hammondsville deposit.
- (4) The Chester, Vermont, area should be surface-explored to determine if other similar deposits occur. The Carleton Quarry and the Vermont Talc Company quarry should be examined.

RECOMMENDATIONS

Excluding the matters of reserves and economics, the Hammondsville deposit appears preferable mineralogically inasmuch as the talc platelets are flatter and more equidimensional, and rutile and carbonate are known to be removable by beneficiation. Tremolite, which is present in the Gouverneur product, is a decidedly objectionable needle-like particle.

Very truly yours,

William Lee Smith

William Lee Smith
Principal Geologist

WLS:cw

TriPLICATE

cc: Dr. W. H. Lycan
Mr. J. N. Masci
Mr. C. V. Swank

Exhibit 32

Battelle Memorial Institute

505 KING AVENUE COLUMBUS I, OHIO

August 25, 1961

Mr. W. H. Ashton
Research Department
Johnson and Johnson
New Brunswick, New Jersey

Dear Mr. Ashton:

The following constitutes an evaluation of the exploration work done to date on the Hammondsburg talc deposit.

Surface holes 1, 2, 3, and 4 indicate the probable reserves of talc to be in excess of one-half million tons of ore, and the possible reserves to be greater.

The cores from holes 1 and 2 were examined and discussed with Mr. P. Bleser. They show the following:

	<u>Hole 1, feet</u>	<u>Hole 2, feet</u>
Overburden	0- 14	0- 2
Schist	14-104	2-165
Talc	104-120	165-169
"Cinder"	120-125	169-179
Talc	125-127	179-200
Schist	127-150	200-212

The talc body appears to thin down-dip from the mine. The talc from hole 1 is slightly darker than current production due to more abundant opaques and greenish gangue components. However, the talc is superior to the off color "unsatisfactory" ore previously obtained from the corresponding part of the mine. The major section of ore from hole 2 is about current grade. Ore from these thinner parts of the ore body will necessarily have a higher percentage of dilution by wall rock and border phase ore than that from the thicker parts of the ore body, unless the ore is selectively mined.

The drilling of hole 3 was observed. The core showed the following:

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Mr. W. H. Ashton

-2-

August 25, 1961

Hole 3, feet

Overburden	0- 18
Schist	18-245
Talc (only 2' recovered)	245-249
Schist	249- —

The ore pinches in hole 3 in a position roughly down-dip from a similar pinching in the mine. Whether this thinning represents a continuation of that constriction of the ore seen in the mine, or a pinching-out down-dip, would have to be determined by further exploration. The ore from the center part of the talc section is of good grade; however, due to the poor recovery a composite sample shows a disproportionately high mica and amphibole contamination.

Hole 4 was selected during my recent visit. The results of the drilling were reported to me by Mr. V. Backels, as follows: talc from 147 to 193 feet, and from 203 to 265 feet, none to bottom at 273 feet. This is a total of 108 feet of talc. Eastern Magnesia will send to me specimens from the upper and lower talc zones for petrographic examination. The two zones were reported to have minor textural differences. The ore is reported to be high-grade white talc. Whether or not the platy character of the ore has been maintained in the thickened body is yet to be determined.

Generally speaking, the mineral purity and whiteness would be expected to be maintained or improved along the apparent strike of the ore toward the thicker section, and possibly to decrease down-dip from the mine. The character of the ore at the present heading and in hole 4 is of greater significance to the mineral nature of the deposit as a whole than is the character of the ore in holes 1, 2, and 3. A critical fact is whether or not the platy character of the talc is maintained throughout the greatly thickened section of the ore body.

Specific differences between the ore in the first three holes are not significant since proper sampling of the cores will not be possible until they are split, crushed, and composites are made for experimental work. Grain counts of the typical ore in cores 1, 2, and 3 showed the following range of composition:

Platy talc	60-65%
Nonplaty talc	1-4
Carbonates	30-35
Altered amphiboles	tr-1
Opaques (including pyrite)	tr->2
Chloritized silicates	<1->1
Micas	tr-1
Quartz, feldspar	tr
Rutile, fluorite, zircon, magnetite	0

Ore from hole 4 has not yet been examined.

Battelle Memorial Institute

Mr. W. H. Ashton

-3-

August 25, 1961

A sample of selected talc ore was high-graded from the pile near the roadside. This sample represents current ore without wall rock dilution and should correspond to selectively mined ore. The previous sample which was sent to Denver contained abundant gangue, more closely representing run-of-mine ore. The new sample was taken for comparative purposes, for possible work by Denver Equipment and Mr. Perkins. I have recommended to Mr. W. Magnus that Mr. Perkins repeat his initial beneficiation procedure (in which he obtained a product with a brightness of 84) on this newly obtained selected ore. The new sample of ore will also give Denver a chance to show what they can do with selectively mined ore.

The crystallographic habit of the talc in hole 4 and subsequent holes has yet to be determined. If the ore is uniform, the matter of reserves is no longer a problem. Inasmuch as the mine was flooded during my last visit to Hammondsville, it was impossible to sample the ore for rutile or to sample the current heading and shaft, as had been proposed. It is advisable that I take care of these details when I go to Vermont to study the subsequent cores, the composite sample, and the flotation products. A primary remaining problem is improving the color of the flotation product.

Very truly yours,

Bill

Wm. L. Smith
Principal Geologist

WLS:lb

Exhibit 33

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE
P.O. Box 112
GOLDEN, COLORADO 80401

April 14, 1971

CSMRI Project No. 200534 ✓

Mr. Robert Russell
Johnson & Johnson
Research Division
New Brunswick, NJ 08901

Dear Mr. Russell:

As requested in your letter of April 1, 1971 to Mr. Robert C. Merritt, x-ray diffraction and microscopic analyses have been completed on the two Vermont final product samples. In your letter you stated Sample A (CSMRI No. 15) was produced using the delaminator and that Sample B (CSMRI No. 16) was produced without using the delaminator.

SUMMARY AND CONCLUSIONS

X-ray diffraction and microscopic studies showed the samples differ in some respects. X-ray diffraction studies indicated a trace of tremolite-actinolite in CSMRI Sample 16; no tremolite-actinolite was noted in the x-ray diffractogram of CSMRI Sample 15.

Microscopic studies of the two samples indicated:

1. Sample 16 (undelaminated) was slightly finer grained than Sample 15, possibly due to preferential liberation during the delamination process followed by preferential flotation of large talc plates.
2. Both samples contained some needle-like particles whose refractive indices were above the refractive index of the 1.600 oil used. These particles were tentatively identified as tremolite-actinolite. Sample 16 (undelaminated) contained a noticeably larger amount of these particles than did Sample 15 (delaminated). Again, this effect is possibly due to preferential floatability of large talc plates as opposed to reground tremolite-actinolite needles.
3. The platy content of Sample 15 (delaminated) was slightly higher than the platy content of Sample 16 (undelaminated).

Mr. Robert Russell

Page 2

April 14, 1971

4. The fibrous content of Sample 16 (undelaminated) was slightly higher than the fibrous content of Sample 15 (delaminated).
5. Overall, Sample 16 (undelaminated) looked as if it had been ground more than Sample 15 (delaminated).

RESULTS AND DISCUSSION

The results of the x-ray diffraction study are shown in Figure 1. As may be noted from the tracings of the diffractograms, the mineralogical composition of both samples is essentially the same. The only difference noted was the trace amount of tremolite-actinolite in Sample 15 (delaminated).

Microscopic analyses of the as-received samples yielded the following results (all values are visual estimates):

CSMRI Sample No.	Platy %	Foliated %	Fibrous %	F.G.A. %	CO ₃ %	Dark Opaque %	Tremolite- Actinolite %	mm			
								>0.1	0.1-0.05	0.05-0.01	<0.01
15	95	3	2	<1	tr	<1	tr	<1	50	40	10
16	92	3	4	<1	tr	<1	1	<1	40	40	20

Photomicrographs of these two samples are shown in Figure 2.

The microscopic study indicated Sample 16 (undelaminated) was slightly finer grind than Sample 15, contained a slightly smaller percentage of platy material than Sample 15, and contained a slightly larger amount of fibrous material than did Sample 15. Some needle-like particles were noted in both samples; Sample 16 contained a noticeably larger amount of these needles. These needle-like particles had refractive indices above that of the refractive index of the immersion oil used (1.600). They were tentatively identified as tremolite-actinolite. The x-ray diffraction study tended to substantiate this identification.

Mr. Robert Russell

Page 3

April 14, 1971

Two anomalies are notable among the foregoing results. These anomalies are:

1. The production of a relatively coarse-grained product from feed that has been processed through the delaminator - a process that is known to result in some size reduction.
2. The appearance of a significantly larger amount of liberated tremolite-actinolite in the undelaminated product.

Several explanations are possible for the first anomaly.

- a. The possibility that the delaminator is producing a relatively large quantity of thin talc plates from a relatively few talc books. These books of plates would have appeared as single plates before delamination.
- b. The inherent higher floatability of large plates as opposed to fines.

The second anomaly can also be explained by the inherent floatability of large plates and fibers as opposed to fines. Preliminary (unreported) studies of unbeneficiated ore taken before and after the delaminator indicate that the delaminator significantly grinds fibers to very small size.

Should you have any further questions regarding these samples, please feel free to contact us.

Sincerely,

R. C. Beers

Robert C. Beers
Project Engineer
Metallurgical Division

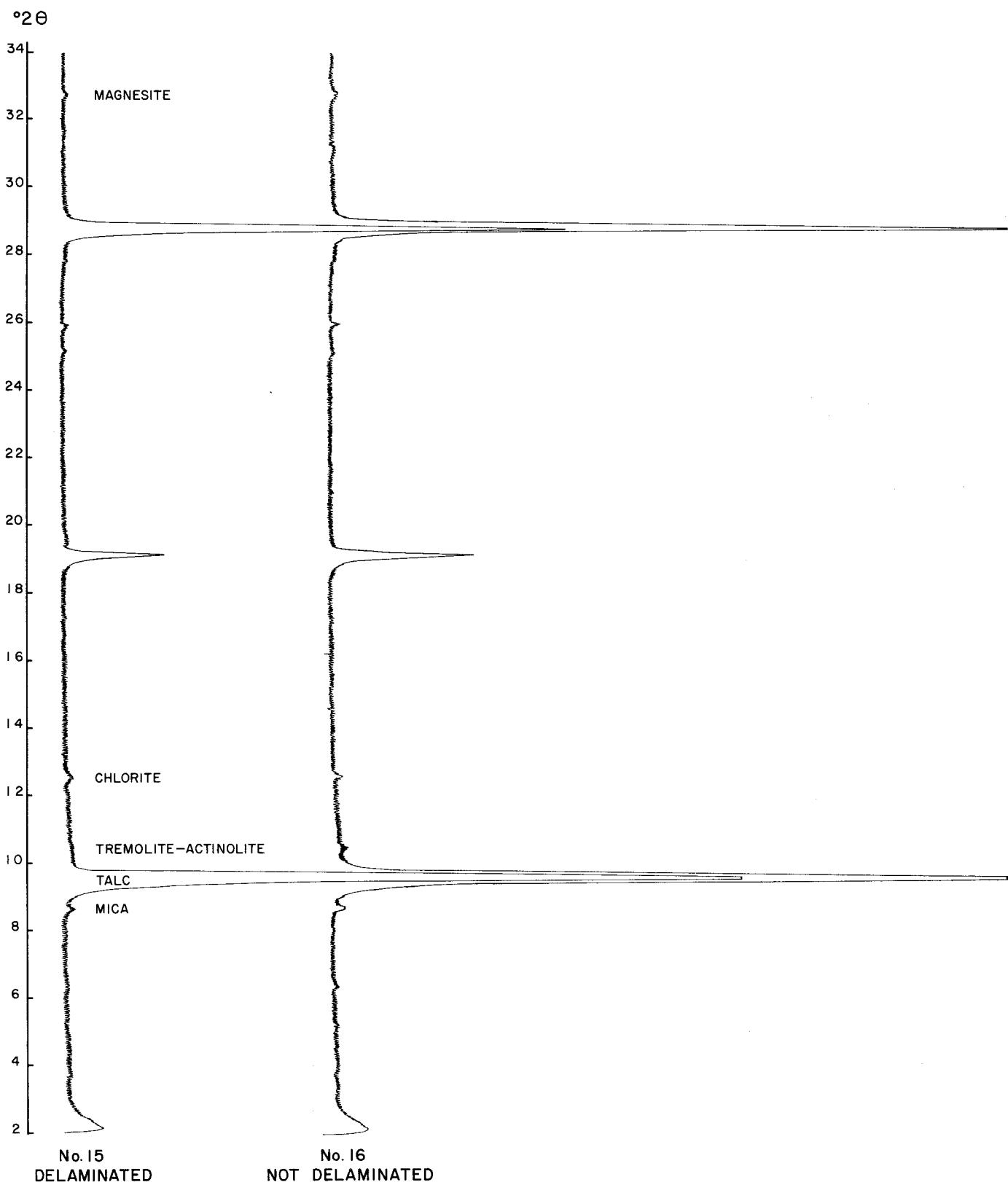
AND

M. G. Pattengill

M. G. Pattengill
Project Engineer
Mining Division

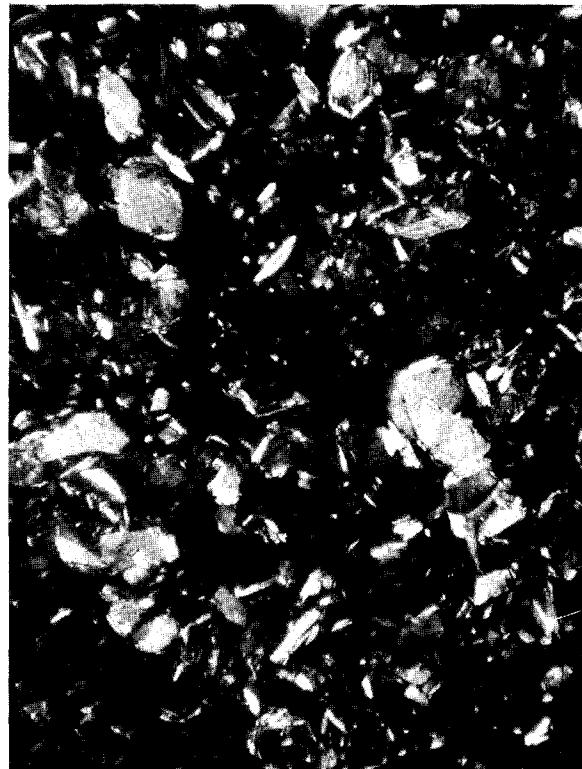
/arh
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FIGURE I
X-RAY DIFFRACTOGRAMS OF TWO SAMPLES OF VERMONT TALC
(Cu RADIATION, Ni FILTER, 2° MIN., SCALE FACTOR 1×10^4)





Sample 15, produced using the
delaminator.



Sample 16, produced not using the
delaminator.

Scale
—
0.1 mm

Figure 2. Photomicrographs of two Vermont talc samples.

Exhibit 34



Dr. A. J. Goudie
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

EXAMINATION
OF
JOHNSON AND JOHNSON'S BABY POWDER

Date: 27 October 1972
MA Number: 2546
Copy 1 of 4

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This REPORT.
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walter c. mccrone associates, inc.
2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616

EXAMINATION OF JOHNSON AND JOHNSON'S BABY POWDER

Summary

Two samples of Johnson and Johnson's Baby Powder, batch number 108T and 109T, which correspond to the samples examined by Professor Seymour Z. Lewin of New York University on behalf of the FDA have been examined by x-ray diffraction, light microscopy, transmission electron microscopy and electron diffraction to determine whether they contain any asbestos minerals.

Both samples contained an insignificant amount of tremolite (< 0.5%). Neither sample contained chrysotile.

Introduction

On behalf of the FDA, Professor Seymour Z. Lewin of New York University is examining a number of commercial talcum powders for the presence of asbestos minerals. Two of the samples which he has examined are samples of Johnson and Johnson's Baby Powder, batch number 108T and batch number 109T. Johnson and Johnson therefore requested Walter C. McCrone Associates to examine samples from the same batches to determine whether they contained any asbestos minerals.

Materials and Method of Conducting Tests

Two samples were submitted, identified as Johnson and Johnson's Baby Powder, batch numbers 108T and 109T.

For x-ray diffraction examination, the samples were examined on a Phillips-Norelco vertical diffractometer using CuK α radiation and a scanning speed of 1° per minute. The dispersion staining technique was used for the light microscopical examination and the electron microscopy-electron diffraction examination was carried out using procedures previously described (MA report 2330-1; dated 10 August 1971).

walter c. mccrone associates, inc.

Results

X-ray Diffraction

The diffractograms were carefully examined in the vicinity of the major peaks of chrysotile and tremolite. Neither mineral was present. The presence of peaks in the vicinity of 12.0-12.5° 2θ , the region in which one of the principal lines of chrysotile may be found, was correlated with peaks in the vicinity of 6° 2θ and are thus attributable to chlorites. No significant peaks were observed in the 24° region which would be required were chrysotile present.

Light Microscopy

Using the dispersion staining technique and a liquid of refractive index 1.550, the samples were examined for chrysotile particles and fibers, but none could be found. Using a similar technique with a liquid of refractive index 1.605, the samples were similarly examined for the presence of tremolite and a few individual crystals were found, some rod shaped. The total tremolite content of the two samples would be approximately 0.5% for 108T and about 0.2-0.3% for 109T.

Electron Microscopy and Electron Diffraction

Several electron microscope grids from both samples were examined in their entirety and although some fibers were observed these were shown by electron diffraction to be shards of talc or rolled talc. No chrysotile fibers were found.

Conclusion

A detailed examination of two samples of Johnson and Johnson's Baby Powder, batch numbers 108T and 109T has shown this material to be substantially free of asbestos minerals. A few tremolite rods were observed in both samples at a level less than 0.5%. No chrysotile has been detected.

Respectfully submitted,

Ian M. Stewart

Ian M. Stewart
Manager, Electron Optics Group

Exhibit 35

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE
124547

P.O. Box 112

GOLDEN, COLORADO 80401

TO W. H. Ashton DATE February 26, 1973
FROM W. P. Reid and W. T. Caneer PROJECT NO. C10704
SUBJECT Mineralogical Examination of Five Talc Samples

In compliance with your request, mineralogical studies were made on the following talc samples:

30-71-S
30-B-71-S
30-C-71-S
32-71-S
34-71-S

The purpose of these studies was to determine the mineralogy of these samples with an emphasis on the occurrence of any asbestos type minerals. X-ray Diffraction and microscopic studies were made on as-received samples, heavy liquid separates, and on acid leached residues.

SUMMARY

The following table shows the nature and relative abundance of minerals in each sample.

Sample	Mineralogy
30-71-S	Major (>40%) talc $[\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2]$ Moderate (20-40%) magnesite (MgCO_3) Trace (<1%) chlorite Trace calcite (CaCO_3) and dolomite $[\text{CaMg}(\text{CO}_3)_2]$ Trace opaques (Fe oxides, etc.) Slight trace (<0.1%) tremolite-actinolite (Ca-Mg-Si-O-OH)
30-B-71-S	Major talc Moderate magnesite Trace dolomite and calcite Trace opaques Slight trace chlorite Slight trace tremolite-actinolite

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE

124548

P.O. Box 112

GOLDEN, COLORADO 80401

TO W. H. Ashton DATE February 26, 1973
FROM W. P. Reid and W. T. Caneer PROJECT NO. C10704
SUBJECT Mineralogical Examination of Five Talc Samples
Page 2

Sample	Mineralogy
30-C-71-S	Major talc Minor (5-20% magnesite Very minor (1-5% chlorite) Trace calcite and dolomite Slight trace opaques
32-71-S	Major talc Moderate magnesite Very minor chlorite and possible serpentine $[\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4]$ Trace calcite and dolomite Very minor opaques Slight trace anthophyllite (?) $[(\text{Mg}_1\text{Fe})_7(\text{Si}_8\text{O}_{22}(\text{OH})_3)]$
34-71-S	Major talc Minor magnesite Very minor chlorite Very minor dolomite Trace mica $[\text{K-Al-Si-O-OH}]$ Trace opaques

Relative to possible asbestos type minerals the above table shows that Samples 30-71-S and 30-B-71-S contain slight traces of tremolite-actinolite minerals. Sample 32-71-S is suspected to contain a very minor amount of serpentine which may be chrysotile. In addition a slight trace of possible anthophyllite was observed in this sample. It is recommended that more studies be made on greater quantities of Sample 32-71-S to confirm the presence of these minerals.

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE

P.O. Box 112
GOLDEN, COLORADO 80401

TO W.H. Ashton
FROM W.P. Reid and W.T. Caneer
SUBJECT Mineralogical Examination of Five Talc Samples
Page 3

DATE

PROJECT NO.

February 26, 1973C10704RESULTS AND DISCUSSION

Each sample of ground talc ore was separated into the following fractions by centrifuging in heavy liquids: specific gravity less than 2.90; specific gravity greater than 2.90. After making an X-ray diffractogram of the greater than 2.90 specific gravity fractions, the sample was leached with 1:1 HCl to remove magnesite. The insoluble residue was then examined for amphiboles under the petrographic microscope. The weight percent in the various fractions as well as the mineralogy of each fraction is shown in the following table.

Sample	Weight, %	Mineralogy
30-71-S	100.0	
Specific gravity > 2.90	74.5	Major (>40%) talc Very minor (1-5%) dolomite Trace (<1%) chlorite Trace calcite and magnesite
Specific gravity < 2.90	25.5	Major magnesite Very minor talc Trace dolomite and calcite
HCl leach residue	0.4	Major opaques (magnetite, etc.) Major talc Slight trace (<0.1%) tremolite-actinolite
30-B-71-S	100.0	
Specific gravity < 2.90	76.4	Major talc Minor (5-20%) magnesite Trace dolomite Slight trace chlorite

Case 3:16-md-02738-MAS-RLS Document 16133-2 Filed 12/22/20 Page 157 of 225 PageID:
COLORADO SCHOOL OF MINES RESEARCH INSTITUTE
P.O. Box 112
GOLDEN, COLORADO 80401

TO W.H. Ashton DATE February 26, 1973

FROM W.P. Reid and W.T. Caneer PROJECT NO. C10704

SUBJECT Mineralogical Examination of Five Talc Samples
Page 4

Sample	Weight, %	Mineralogy
30-B-71-S (cont'd) Specific gravity >2.90	23.6	Major magnesite Very minor talc Trace calcite
HCl leach residue	0.4	Major talc Major opaques (magnetite, sulfide, etc.) Slight trace tremolite-actinolite
30-C-71-S Specific gravity <2.90	100.0 90.5	Major talc Minor magnesite Very minor chlorite Trace dolomite
Specific gravity >2.90	9.5	Major magnesite Minor talc and dolomite Trace calcite
HCl leach residue	<0.1	Major opaque Minor talc
32-71-S Specific gravity <2.90	100.0 58.4	Major talc Very minor magnesite Very minor chlorite and serpentine (?) Trace calcite
Specific gravity >2.90	41.6	Major magnesite Very minor talc Trace dolomite Slight trace calcite

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE

P.O. Box 112
GOLDEN, COLORADO 80401

TO W. H. Ashton DATE February 26, 1973
FROM W. P. Reid and W. T. Caneer PROJECT NO. C10704
SUBJECT Mineralogical Examination of Five Talc Samples
Page 5

Sample	Weight, %	Mineralogy
32-71-S (cont'd) HCl leach residue	2.5	Major talc Minor opaques Slight trace anthophyllite (?)
34-71-S Specific gravity <2.90	100.0 80.9	Major talc Very minor magnesite Very minor chlorite
Specific gravity >2.90	19.1	Major magnesite Minor dolomite Trace mica
HCl leach residue	0.4	Major opaques Minor talc

In an attempt to verify the presence of serpentine in Sample 32-71-S X-ray diffraction step scans were made over the critical diffraction peaks of serpentine. Figure 1 shows the results of the step scanning. Since both chlorite and serpentine have diffraction peaks in the 7 Å region, it is possible to confuse these minerals based only on a 7 Å peak. However, a corresponding peak in the 14 Å region verifies the presence of chlorite. Curve 1 (Figure 1) represents a step scan across the 7 Å region for the as-received Sample 32-71-S. As may be seen two peaks occur at 7.1 Å and 7.3 Å. In order to determine if these peaks represent chlorite or possibly serpentine, step scans were made across the 14 Å region. If these 7.1 Å and 7.3 Å peaks represent chlorite, then there should be corresponding peaks at 14.2 Å and 14.6 Å. Curve 1A is a step scan across the 14 Å region. At 14.2 Å peak occurs which corresponds to the 7.1 Å peak. These 2 peaks represent chlorite. However, no 14.6 Å peak is present which suggests that the 7.3 Å represents serpentine-not chlorite.

COLORADO SCHOOL OF MINES RESEARCH INSTITUTE
124552

P.O. Box 112

GOLDEN, COLORADO 80401

TO W. H. Ashton DATE February 26, 1973
FROM W. P. Reid and W. T. Caneer PROJECT NO. C10704
SUBJECT Mineralogical Examination of Five Talc Samples
Page 6

Since chlorite has a specific gravity greater than 2.65 and serpentine has a specific gravity less than 2.65, step scans were made on the fraction of Sample 32-71-S which has a specific gravity less than 2.65. The less than 2.65 specific gravity fraction was 0.4 weight percent of the total sample. If serpentine is present the 7.3 Å peak should be enhanced and the 7.1 Å chlorite peak should be diminished. Curve 2 represents a step scan across the 7 Å region of the fraction with a specific gravity less than 2.65. As may be seen the 7.3 Å peak is enhanced and the 7.1 Å peak is diminished relative to the as-received sample. Curve 2A (step scan across the 14 Å region) shows no 14.6 Å peak.

Based on the above step scanning data there is good reason to suspect that serpentine is present in Sample 32-71-S. It is possible that other minerals such as kaolinite or a very iron rich chlorite could give similar data. However, based on the geological and chemical factors associated with this sample, it is more probable that chlorite present will be magnesium-rich rather than iron rich and that serpentine is a more likely occurrence than kaolinite.

Microscopic examination of the fraction with a specific gravity less than 2.65 revealed very minor (1%) amounts of possible serpentine fibers. Identification of the serpentine was facilitated by staining with 1% iodine in glycerine.

It is recommended that further work be done on this sample.

Exhibit 36

Johnson & Johnson

J

NEW BRUNSWICK, N. J.

June 6, 1973

Dr. F. D. Pooley
Department of Mineral Exploitation
University College
Newport Road
Cardiff, Wales
CF2 1TA
GREAT BRITAIN

Dear Fred,

Following the request of Tom Shelley, I will be sending you shortly, 26 ten-gram split production samples of Vermont Talc to be put through the British Toilet Preparations Federation density concentration technique. After I receive the proposed procedure for this density concentration technique from Bob Dean, I will have the same samples put through this procedure by us. Also, in the same shipment will be 3 three-hundred gram samples of production Vermont Talc for your study to remove tremolite.

Best regards.

Sincerely yours,

F. Robert Rolle

F. Robert Rolle

ab

cc: Mr. W. H. Ashton
Dr. M. H. Goodman
Dr. A. J. Goudie
Dr. W. Nashed
Dr. D. R. Pettersen
Dr. T. H. Shelley

RECEIVED

JUN 6 1973

W. NASHED
JOHNSON & JOHNSON

AIR
MAIL

Exhibit 37



walter c. mccrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

11 February 1974

Dr. F. R. Rolle
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Dr. Rolle:

We have completed the analyses of your samples S5-888, T-280 and S1-1028-72831, as per your request.

We found chrysotile only in sample S5-888, T-280 and tremolite in neither of the samples. As a rough estimate, good to about an order of magnitude, I would place the amount of chrysotile present at $\leq .0005\%$.

Representative photomicrographs are included with this report. Thank you for consulting McCrone Associates. If you have any questions concerning any aspect of this work, please feel free to call.

Yours sincerely,

A handwritten signature in black ink, appearing to read "Richard Shimp".

Richard Shimp
Electron Microscopist

RS:fe
Enclosures
Ref: 2546

Exhibit 38



walter c. mccrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

10 April 1974

Mr. R. S. Russell
Johnson and Johnson
501 George Street
New Brunswick, New Jersey 08901

Dear Mr. Russell:

Using the transmission electron microscope we have examined four (4) samples of talc designated as A, UDSA, UDSB and AC.

Sample AC contained one small fiber of chrysotile about $1 \mu\text{m}$ in length. There was also some organic film which bubbled and decomposed in the electron beam. This film apparently is soluble in the isopropyl alcohol used to disperse the samples and on drying forms a thin film covering the specimen grid. Most of the sample was clean with not much rolled talc or talc shards.

Sample UDSB also contained one fiber of chrysotile. Again this fiber was small, in the size range of $1 \mu\text{m}$. There were some talc shards in this sample but most of it was flaky quality talc.

Sample UDSA was mostly platy talc with no fibers and no talc shards.

Sample A contained a high amount of talc shards and fibrous or ribbon-like talc. I would estimate the percentage of this fibrous ribbon-like talc and talc shards as 1-5%. The plates were small and there was also an organic film present. Electron micrographs and diffraction patterns are included with this report. If there are any questions on this report or the data contained herein, please feel free to contact me.

Very truly yours,

Gene R. Grieger
Gene R. Grieger
Research Physicist

GRG:fe
Enclosures
Ref: 2546

Exhibit 39

ATTACHMENT 6

Mr. Vernon Zeitz
Windsor Minerals Company
Windsor, Vermont 05089



EXAMINATION OF TALC SAMPLES

ARGONAUT ORE BODY

Date: 24 April 1974

MA Number: 3295

Copy \ of 7

walter c. mccrone associates, inc.
2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616

Examination of Talc Samples

Argonaut Ore Body

SUMMARY

An intensive examination has been made by x-ray diffraction and electron microscopy of 38 core samples taken from a new ore body which Windsor Minerals, Inc. are contemplating exploiting. The examination was undertaken to determine the mineralogical content of the core samples and, in particular, whether or not there was any significant content of asbestos-form minerals in the ore body. For comparison, three core samples from the current ore body were also examined.

The majority of the samples showed no evidence of any asbestos-form minerals present and, of the 15 samples that did show an asbestos-form mineral, only one exceeded an estimated level of approximately 0.0005%. It is anticipated that beneficiation of the ore would significantly reduce these low levels and that, therefore, the beneficiated ore would prove to be free of any asbestos-form mineral. It is concluded that the ore body is of suitable quality for the manufacture of high grade cosmetic and toiletry products.

INTRODUCTION

In connection with the assessment of a new talc ore body which Windsor Minerals, Inc. are developing, they requested the assistance of Walter C. McCrone Associates in determining whether or not the ore body would prove to be contaminated by asbestos minerals which might prove to be a potential health hazard. An intensive investigation of 38 core samples taken from the ore body and three samples taken from their currently used ore was therefore carried out using the techniques of x-ray diffraction and transmission electron microscopy combined with electron diffraction.

This report records the results of this examination.

MATERIALS AND METHOD OF CONDUCTING TESTS

Forty-one samples were received from Windsor Minerals, Inc. These were identified as ore body core samples and bore a number which corresponded to their location in the ore bodies.

A portion of each sample was prepared for x-ray diffraction and was examined in a Philips vertical diffractometer using CuK α radiation at a scanning speed of 1° per minute.

Another portion from the sample was prepared for electron microscopy by suspending in isopropyl alcohol and transferring these suspensions to an electron microscope support grid which had previously been coated with a carbon support film. The resulting preparation was examined in the JEM 200 transmission electron microscope using an accelerating voltage of 150 kV. The sample was scanned at a magnification of approximately 25,000X and electron diffraction was carried out on those fibers which were suspect, that is, those fibers which were not readily identifiable as rolled talc or talc splinters and shards. Representative electron micrographs were taken of all samples and also of suspect fibers.

RESULTS

The results of the x-ray diffraction examination and of the electron microscopical examination are summarized in Tables 1 and 2.

X-Ray Diffraction Examination

The principal contaminant minerals found in the core samples are magnesite, chlorites and quartz. The magnesite level is generally high, ranging between about 20% to over 60% although it is difficult to accurately estimate the magnesite content due to preferred orientation effects in some cases enhancing the talc signal. In one sample, however, sample 6-R-72, 179-184, the magnesite content was very low, of the order of 2-3%. In ten of the samples a shorter diffractometer trace was used which did not allow quantitation of the magnesite content.

No attempt has been made to identify which chlorites are present in these samples: the chlorite group of minerals is comprised of some twenty to thirty minerals which are closely related in their interatomic spacings and their specific identification is not relevant to the present problem.

In no instance was any asbestos or potentially asbestiform mineral identified by x-ray diffraction, the limit of whose sensitivity is approximately $\frac{1}{2}$ to 1% for the amphibole minerals and probably slightly higher than this for chrysotile asbestos..

Electron Microscopical Examination

All the samples were examined extensively in the electron microscope concentrating attention on the fibrous components of the samples. In general, all the samples showed a very good, clean, platey talc such as illustrated typically by plates 3538 and 3545 accompanying this report. There were, of course, as in all talc samples, some apparently fibrous components and, in most instances, these are pieces of rolled talc or talc

shards and fronds, resulting from splitting of the talc plates. As the feature of particular interest was the asbestiform minerals which might be present in the material, our attention was focused on these and thus the photographic documentation accompanying this report consists almost exclusively of representations of this small fibrous fraction. As will be seen from Table 2, only two samples (2R-72, 232-257 and 53-4-68, 512½-576) showed a level of asbestos above 0.0005%, the actual figures being 0.007% and 0.001%, respectively, for chrysotile asbestos plus approximately 0.0001% of fibrous tremolite. Excluding these samples, the remaining samples which showed asbestiform fibers are exhibiting levels which are no higher than has been seen in a raw composite used to manufacture a finished product. The levels of chrysotile observed in the two high samples is only an order of magnitude above this and would presumably be reduced considerably by your beneficiating process.

CONCLUSIONS

The examination of 41 core samples, 38 of them from a new talc ore body, using the techniques of x-ray diffraction, electron microscopy and selected area electron diffraction have shown that, even prior to beneficiation, this material is of extremely high grade, substantially asbestos free, and of a quality which we associate with cosmetic grade talc. In only two samples was a level of chrysotile observed which was higher than 0.0005%. Chrysotile levels of this order of magnitude might well arise during the taking and handling of the samples.

Table 1
X-ray Diffraction Analysis of Talc Ore Core Samples

Sample DDH	Description Designation	Quartz	Carbonate	Chlorites	Asbestiform Minerals
1-R-72	87 - 155	n.d.	40-50	~3%	n.d.
	155 - 164	n.d.	60-65	2-3%	n.d.
	164 - 176	n.d.	35-40	5-8%	n.d.
	184 - 241	n.d.	30-35	~6%	n.d.
2-R-72	131 - 167	n.d.	35-45	3-5%	n.d.
	183 - 232	n.d.	30-40	3-5%	n.d.
	232 - 257	n.d.	50-60	~3%	n.d.
	257 - 268	n.d.	30-40	3-4%	n.d.
3-R-72	51 - 62	n.d.	n.a.	10-15%	n.d.
	158.5 - 170	n.d.	n.a.	3-5%	n.d.
	174.5 - 190	n.d.	50-60	5-8%	n.d.
	229 - 240	n.d.	45-55	10-15%	n.d.
6-R-72	92 - 111	n.d.	20-30	2-3%	n.d.
	112 - 133	trace	40-50	3-4%	n.d.
	147 - 165	n.d.	30-35	~2%	n.d.
	166 - 176	?	25-35	8-10%	n.d.
	179 - 184	n.d.	2-3	10-15%	? chrysotile
8-R-72	49 - 89	n.d.	n.a.	3-5%	n.d.
	98 - 115	n.d.	n.a.	2-3%	n.d.
	136.5 - 141	n.d.	n.a.	3-5%	n.d.
	160.5 - 192	n.d.	20-30	3-5%	n.d.
	207 - 212	n.d.	n.a.	2-4%	n.d.
9-R-72	38 - 92	n.d.	40-50	3-5%	n.d.
	93 - 103	n.d.	30-35	1-2%	n.d.
	205 - 260	n.d.	40-50	3-4%	n.d.
	267 - 275	n.d.	30-40	1-2%	n.d.
11-R-72	152.5 - 169	n.d.	n.a.	5-8%	n.d.
	177 - 193	n.d.	n.a.	3-5%	n.d.
	196 - 219	n.d.	n.a.	5-8%	n.d.
	224 - 236	n.d.	n.a.	5-8%	n.d.
18-R-73	183 - 214	n.d.	30-35	~3%	n.d.
	277 - 297	n.d.	50	2-3%	n.d.
	297 - 304	n.d.	40-50	~5%	n.d.
19-R-73	208 - 237	n.d.	30-35	~3%	n.d.
	237 - 248	n.d.	30-40	2-3%	n.d.
	248 - 257	n.d.	20-30	2-3%	n.d.
	257 - 277	n.d.	20-30	2-3%	n.d.
	278 - 294	n.d.	50-60	2-3%	n.d.
10-H-67	{366 - 370}	n.d.	30-40	1-3%	n.d.
	{374 - 402}				
36-H-67	399 - 466	n.d.	50-60	8-10%	n.d.
53-H-68	512.5 - 576	trace ??	10-20	5-8%	n.d.

*n.a. = not analyzed

n.d. = not detected

Table 2
Electron Microscopic Analyses of Talc Ore Core Samples

Sample DDH	Description Designation	Chrysotile	Amphibole
1-R-72	87 - 155	n. d.	n. d.
	155 - 164	n. d.	n. d.
	164 - 176	0.0002%	n. d.
	184 - 241	n. d.	n. d.
2-R-72	131 - 167	0.0001%	n. d.
	183 - 232	0.0001%	n. d.
	232 - 257	0.007%	0.0001%
	257 - 268	0.0001%	n. d.
3-R-72	51 - 62	n. d.	n. d.
	158.5- 170	n. d.	n. d.
	174.5- 190	n. d.	n. d.
	229 - 240	n. d.	n. d.
6-R-72	92 - 111	0.0003%	n. d.
	112 - 133	0.0001%	0.0001%
	147 - 165	n. d.	n. d.
	166 - 176	0.0002	n. d.
	179 - 184	0.0004%	0.0001%
8-R-72	49 - 89	n. d.	n. d.
	98 - 115	n. d.	n. d.
	136.5- 141	n. d.	n. d.
	160.5- 192	n. d.	n. d.
	207 - 212	n. d.	n. d.
9-R-72	38 - 92	0.0002%	n. d.
	93 - 103	n. d.	n. d.
	205 - 260	0.0005%	n. d.
	267 - 275	0.0001%	n. d.
11-R-72	152.5- 169	n. d.	n. d.
	177 - 193	n. d.	n. d.
	196 - 219	n. d.	n. d.
	224 - 236	n. d.	n. d.
18-R-73	183 - 214	n. d.	n. d.
	277 - 297	n. d.	n. d.
	297 - 304	n. d.	n. d.
19-R-73	208 - 237	n. d.	n. d.
	237 - 248	0.0003%	n. d.
	248 - 257	n. d.	n. d.
	257 - 277	n. d.	n. d.
	278 - 294	0.0004%	n. d.
10-H-67	{366 - 370} {374 - 402}	n. d.	n. d.
36-H-67	399 - 466	n. d.	n. d.
53-II-68	512.5- 576	0.001%	0.0001%

It is our conclusion, based on the platyness of the material and its freedom from asbestosiform minerals, that the ore body would be suitable for use in high quality cosmetic and toiletry products.

Respectfully submitted,

Richard Shimps/12

Richard Shimps
Research Chemist

Ian M. Stewart

Ian M. Stewart
Manager, Electron Optics Group

RS:IMS:smg

walter c. mccrone associates, inc.

Exhibit 40

April 27, 1973

B128-04

Scanned

Microscopic Examination of Johnson's Baby Powder

For amphibole (tremolite-actinolite)

<u>Lot Numbers:</u>	285S	10/11/72
	307R	11/2/72
	068Z	3/9/73
	086Z	3/26/73

Petrographic optical microscopy revealed "trace" amounts of amphibole in each of the above samples.

Based on the numbers of particles scanned, we estimate "trace" amounts to be .001 to .01% by weight.

Description of Particles:

Shape: Prismatic, columnar, parallel-sided rods.

Size: From 20 x 4 microns to 200 x 30 microns.

Identity: The optical properties of the particles are closer to actinolite than tremolite.

Remarks: In several of the larger particles, the amphibole was observed to be intrinsically attached to a talc particle.

Exhibit 41



Windsor Minerals Company
Windsor, Vermont 05089

**EXAMINATION OF TALC ORES AND PRODUCTS:
BENEFICIATION PROCESSES**

Date: 8 May 1974

MA Number: 3295

Copy of

walter c. mccrone associates, inc.
2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616

EXHIBIT
J&J-66

Examination of Talc Ores and Products: Beneficiation Processes

I. INTRODUCTION

In connection with the development of improved beneficiation processes, Windsor Minerals, Inc. have requested Walter C. McCrone Associates, Inc. to carry out an extensive examination of 6 samples of talc ores and talc products produced from these ores using the methods of light microscopy and transmission electron microscopy. This report records the results of these examinations.

II. MATERIALS AND METHODS OF CONDUCTING TESTS

Six samples were received identified as follows:

66-A-ore
66-A-product
66-U-ore
66-U-product
66-AC-ore
66-AC-product

For light microscopical examination the samples were prepared by mounting a small portion of the talc powder in Aroclor 5442 on a glass slide and were examined on a Zeiss G.F.L. polarizing microscope at magnifications in the range of 50X to 400X. Photomicrographs were taken of the samples showing representative areas at magnifications of 55X and 140X on Kodachrome A color film, using slightly uncrossed polars (12°). Accompanying this report are prints prepared from these color transparencies at magnifications of 165X and 420X.

For examination by transmission electron microscopy, samples were prepared as follows: Approximately 0.1 g of powder was suspended in about 25 ml of isopropyl alcohol, shaken manually, then held in an ultrasonicator

for a few minutes. During this time some of the larger flakes settle toward the bottom of the tube. The remainder of the suspension was immediately placed on a standard 3 mm diameter transmission electron microscope grid which had previously been coated with a carbon support film and which was supported on a glass slide. This glass slide was then transferred to a hot plate where the isopropanol carrier was evaporated and the dried specimen grid was examined in a JEM-200 transmission electron microscope at 150 kV. A considerable number of grid squares was examined for each sample. Representative electron micrographs were taken and prints are included with this report.

III. RESULTS

Sample 66-A-ore — Light microscopical examination showed that this was predominately platy talc with approximately 40% carbonate present. Talc plates ranged in size from a few micrometers to up to 300-400 μm across but showed a very uniform distribution of thicknesses with very flat plates and little folding or buckling of the talc. Some talc rolls were present but they probably represented less than 5% of the total sample. A few quartz grains were also identified but no identifiable asbestos was found.

Electron microscopical examination largely confirmed these findings showing the sample to be predominately platy material ranging from small to quite large platelets with some rolled talc present. There was very little in the way of other inorganic fibers present, although one fiber, probably tremolite, was found with an overall length of 8.6 μm and a width of 1.2 μm . No chrysotile was detected in this sample. The blocky material present in this sample which gave a hexagonal electron diffraction pattern is identified on the basis of the light microscopy as the carbonate phase. A few other fibrous or rod-shaped particles observed appear to be talc in one form or other; i.e., rolled talc or talc shards and were unmistakably talc on the basis of their diffraction patterns.

Sample 66-A-product — Light microscopical examination of this material showed that removal of the carbonate by the beneficiation process was complete and that the material was a good, platy talc. The plates still showed good uniformity of thickness although rumpling was evident in some of the plates, probably as a result of deformation during processing. No identifiable asbestos was detected by light microscopy. The size range of the talc present was similar to that exhibited by the ore. Electron microscopical examination confirmed the results of light microscopy, the sample is predominately platy material, the blocky material found in the ore was no longer present and there was very little in the way of a fibrous fraction. No fibrous amphiboles or other asbestos minerals were detected in this sample by electron microscopy.

Sample 66-U-ore — Light microscopical examination showed this was predominately platy talc similar to the 66-A-ore, but showing more evidence of damage to the talc plates; that is, there were more wrinkled plates and these plates were also less uniform in thickness than in the 66-A-ore. The fibrous content of this sample was also slightly higher than in the 66-A-ore but was still probably less than 5% of the total. These fibrous forms were talc rolls and shards. No asbestos was identifiable by light microscopy.

Electron microscopical examination again confirmed the light microscopical examination showing platy material to be predominant, ranging from very small to very, very large plates, with a quite continuous distribution of sizes. Rolled talc and broken platelets with a fibrous aspect ratio were also observed as were the blocky fragments of carbonate. Overall, this sample had a much better appearance than the 66-A-ore. There was no evidence by electron microscopy of fibrous asbestos forms in this sample.

Sample 66-U-product — Light microscopical examination showed that removal of the carbonate by the beneficiation process was not quite complete with up to 1% of the carbonate still present. There was, however, no evidence

of increased damage to the talc plates although there were more talc shards and rolls present than in the 66-A-product. No asbestos minerals were observed by light microscopy.

Electron microscopical examination showed talc ribbons and rolled talc present but the sample mainly consisted of medium to moderately large size platy material with very few inorganic fibers. One very small fiber was present which resembled chrysotile but this could not be confirmed by electron diffraction. No asbestiform amphiboles were observed.

Sample 66-AC-ore — Light microscopical examination indicated that this sample was more similar to 66-U than to 66-A, although the fibrous content was probably slightly higher than A or U, but, again, this fibrous content consisted entirely of talc rolls and shards. Some rumpling of the talc plates was evident and some chlorite grains were observed. No asbestos minerals were detected by light microscopy.

The electron microscopical examination showed that this ore consists, again, mainly of platy material with only a few fibers and some rolled talc present. Eight chrysotile fibers were found in this sample, however, their lengths were all less than 1/3 of 1 μm . No asbestiform amphiboles were observed

Sample 66-AC-product — Light microscopical examination showed the beneficiation process had successfully removed all the carbonates and that it had also either broken down or removed many of the large talc plates (those above about 300-400 μm) and seems to have removed most of the very small material, giving the product a narrower range of sizes than the ore. The fibrous talc content, however, was still probably higher than that of the 66-A or 66-U products. No asbestiform minerals were observed by light microscopy.

Electron microscopical examination of this sample showed this was mostly platy material ranging in size from quite small to very large with talc ribbons, rolled talc and talc fibrils also present. Only one chrysotile fiber was

found in this sample; a significant reduction from the level in the ore sample. Again, no asbestiform amphibole minerals were detected.

IV. SUMMARY

An intensive microscopical examination of 6 samples of talc ore and talc products produced by the beneficiation of these ores has been carried out using light microscopy and transmission electron microscopy combined with electron diffraction.

In all 6 samples only one fiber of a possible asbestiform amphibole was observed — in Sample 66-A-ore. Minute chrysotile fibrils were observed in 3 of the samples, in 2 of these, however, Sample 66-U-product and Sample 66-AC-product, only a single fibril was observed and that observed in the 66-U-product could not be confirmed by electron diffraction. At the level of one fiber in a sample it is debatable whether this represents a true chrysotile level in the sample or whether it represents contamination during taking or preparation of the sample.

Sample 66-AC-ore showed 8 chrysotile fibers but in the product from the same ore beneficiation had reduced the chrysotile content to 1 fibril as mentioned above. The chrysotile fiber content of Sample 66-AC-ore represents an estimated chrysotile content of <1-2 ppm, thus, even in the worst case, the level of asbestos contamination present in these ores is minimal. None of the beneficiated products showed any significant asbestos contamination.

ATTACHMENT B

**ASBESTIFORM DEPRESSION
THROUGH THE USE OF
NEW FLOATATION REAGENT SYSTEMS**

-1-

J&J-0083368

INTRODUCTION:

A study was performed at Windsor Minerals to quantify the effectiveness of two new floatation reagent systems in the depression of asbestos minerals in the floatation process. Analysis of the floated products was accomplished using a Millipore TITMC Image Analyzer as the analytical detection device.

CONCLUSIONS:

1. A combination of n-butyl alcohol as a frother along with citric acid as a depressive agent proved to be 20 times as effective as Ultrawet D.S. suppressing asbestos in the final product.
2. Using only n-butyl alcohol as a frother proved to be 7 times as effective as Ultrawet D.S. in suppressing asbestos.
3. Ultrawet D.S. provided only a minimal suppression of asbestos through the floatation process.

EXPERIMENTAL:

Ground ore from the Hammondsburg Mine was "doped" with 1.0% by weight of the fibrous form of anthophyllite which occurs as a rare mineral in the Hammondsburg ore body, and subjected to a series of laboratory floatations using the following reagent systems:

1. Ultrawet D.S.
2. n-butyl alcohol
3. n-butyl alcohol-citric acid.

The products obtained from these laboratory floatations were scanned on a video monitor coupled to an optical microscope, the system having a useful magnification of 500X. Clearly recognizable asbestos mineral anthophyllite was counted and totalized over 100 viewed fields. The number of particles viewed in the 100 fields were totalized by means of a complementary computer interfaced to the system. The numbers obtained by this technique were compared to those obtained by an identical analysis of a standard preparation consisting of a Grade "66" product "doped" with 2.0% by weight of fibrous anthophyllite.

RESULTS:

Table 1 gives the data and calculated numerical relationships devised to indicate the effectiveness of the new reagent systems in the depression of fibrous anthophyllite. These relationships, their value and definitions are as follows:

1. Rejection factor: a relationship derived to indicate the weight rejection of anthophyllite using a given reagent system. This relationship is arrived at by comparing the anthophyllite weight percentage in the floated product to the 2.0% asbestosiform "doped" product which represents a floated material having undergone no rejection of asbestosiforms from the ore to the product.

The Rejection Factor is defined in these experiments for a given floatation reagent as:

$$\frac{2.0}{\text{anthophyllite weight percentage in floated product}}$$

2. Rejection Ratio - This term relates the effectiveness of suppression of asbestosiforms by the alcohol based systems, to the existing Ultrawet D.S. system and is defined as follows:

$$\text{Rejection Ratio} = \frac{\text{Rejection Factor of new reagent system}}{\text{Rejection Factor of Ultrawet D.S. system}}$$

Table I Asbestiform Analysis of Cosmetic Grade Talc Using TMC
Image Analyzer.

	<u>2.0% Asbestiform Containing Product</u>	<u>Ultrawet D.S. Floated Product</u>	<u>N-Butyl Alcohol Floated Product</u>	<u>N-Butyl Alcohol Citric Acid Floated Product</u>
Total Fields Counted	100	100	100	100
Total Fibers Counted	298	37	6	2
Total Particles Counted	10681	9166	11416	10103
Weight Percentage Asbestiform	2.00	.2894	.0377	.0142
Rejection Factor	1.00	6.91	53.08	140.94
Rejection Ratio	--	1.00	7.68	20.39

SUMMARY AND REMARKS:

The data shows a profound influence of the alcohol based reagent system upon the amounts of asbestos forms reporting in the floated product. It is apparent that the system which includes citric acid is more effective than n-butanol alone.

Although the data was accumulated for the specific mineral species, fibrous anthophylite, the same results can be predicted for other fibrous amphibole minerals and chrysotile asbestos found in association with the Hammondsburg ore body whose surfaces expose a substantial concentration of magnesium and hydroxyl groups as reactive sites.

ATTACHMENT C

MINEROLOGY OF ORES, PRODUCTS AND MILL TAILS RE
DIFFERENT FLOTATION REAGENTS

J&J-0083373

TO: WINDSOR MINERALS INC., Windsor, Vermont 05089
FROM: R. C. Reynolds, Jr., Department of Earth Sciences
Dartmouth College, Hanover, N.H. 03755
SUBJECT: Mineralogy of Ores, Products and Mill Tails Re
Different Flotation Reagents

INTRODUCTION:

A study was made of the mineralogy of talc products and mill tails that were produced by the use of three different flotation schemes. The designations and descriptions used in this report are as follows:

Flotation Agent	Designation
Ultrawet	A
Butanol	B
Butanol + Citric Acid	C

In addition, studies were made of the ore that produced each of the products and mill tails.

TECHNIQUE:

Ores, products, and tails were analyzed by x-ray diffraction methods. Copper $\kappa\alpha$ radiation was used and the region $2\theta = 24^\circ$ to $2\theta = 34^\circ$ was scanned. This 2θ region contains important peaks from talc, chlorite, dolomite and magnesite. Examples of runs are shown on Figure 1. The data in Table 1 was obtained by averaging peak heights from three scans of each sample.

J&J-0083374

Materials were studied for amphiboles by means of the heavy-liquid-benzethonium chloride method described in the Windsor Mineral Report of March, 1974. To improve separation and subsequent semi-quantitative estimation of amphibole, product samples were spiked with dolomite and tourmaline, sized 10-40 μ , to better simulate the ores, which behave well in the amphibole separation procedure.

RESULTS:

Figure 1 shows the x-ray diffraction patterns of ores, products, and tails associated with each of the flotation procedures. Peaks are labelled C = chlorite, T = Talc, D = dolomite, and M = magnesite. The results clearly show

- (1) the low chlorite, magnesite, and dolomite in all of the products
- (2) the large amounts of magnesite, dolomite, and chlorite in ores and tails
- (3) the low concentration of talc in tails B and C

Ores A, B and C are similar as are products A, B and C. The only significant difference among the three treatments shows in the tails; those from treatment A (ultrawet) clearly have a much large talc content than do the tails from the butanol or the butanol-citric acid experiment.

Table 1 shows data tabulated from repeated (three times) runs similar to those shown on Figure 1. The values are meaningful only in a relative sense. There appear to be no significant differences among ores A, B and C, and products A, B and C.

The major difference is among the tails, where tails A is clearly much richer in talc than tails B or tails C.

It is concluded that:

- (1) The ores used for the three flotation experiments are very similar or identical in mineralogy
- (2) The products A, B and C are similar except that product A does have a slightly higher chlorite content
- (3) The tails for B and C are similar, but tails A is clearly higher in talc. Hence, the ultrawet flotation agent clearly produced a higher loss of talc to the mill tails than did the butanol or butanol-citric acid reagents

The results from the amphibole separation are somewhat ambiguous because of the difficulties in obtaining reproduceable extractions from the products. However, the tourmaline added to products A and B was recovered to within $\pm 10\%$ for each, giving confidence in the efficiency of the separation. Based on optical estimates from these samples, and separations of the three done without tourmaline, it is concluded that all three products contain essentially similar concentrations of actinolite, and that its absolute concentration lies between 100 and 200 ppm.

CONCLUSIONS:

As a result of the mineralogical studies reported here, the following are concluded:

- (1) Ores A, B and C are essentially identical with respect to their concentrations of magnesite, dolomite, chlorite and actinolite

- (2) Tails B and C are identical with respect to talc, magnesite, dolomite and chlorite, but tails A is significantly richer in talc
- (3) Products A, B and C are essentially identical with respect to their concentrations of magnesite, dolomite and actinolite; Product A contains a somewhat larger quantity of chlorite
- (4) Amphibole separations from products are difficult to achieve quantitatively, but the addition of carbonate and silicate carriers seems promising in eliminating the difficulties

TABLE I

X-RAY DIFFRACTION PEAK RATIOS FROM ORES, PRODUCTS AND TAILS

	Magnesite/Talc	Dolomite/Talc	Chlorite/Talc
Ore A	0.27	0.12	0.070
Ore B	0.23	0.11	0.086
Ore C	0.25	0.11	0.074
Product A	Very low	Very low	0.0100
Product B	Very low	Very low	0.0086
Product C	Very low	Very low	0.0085
Tails A	1.2	0.78	0.28
Tails B	10.9	5.7	2.6
Tails C	12.2	6.0	2.4

J&J-0083378

TABLE 2

ACID INSOLUBLE HEAVY LIQUID RESIDUES FROM PRODUCTS, ORES AND TAILS

	PPM TOTAL	PPM ESTIMATED AMPHIBOLE
Ore A	9000	~3000
Ore B	10,500	~3000
Ore C	8800	~3000
Product A		100-200*
Product B		100-200*
Product C		100-200*
Tails A	34,600	too much chlorite
Tails B	36,100	too much chlorite
Tails C	44,100	too much chlorite

*See text

J&J-0083379

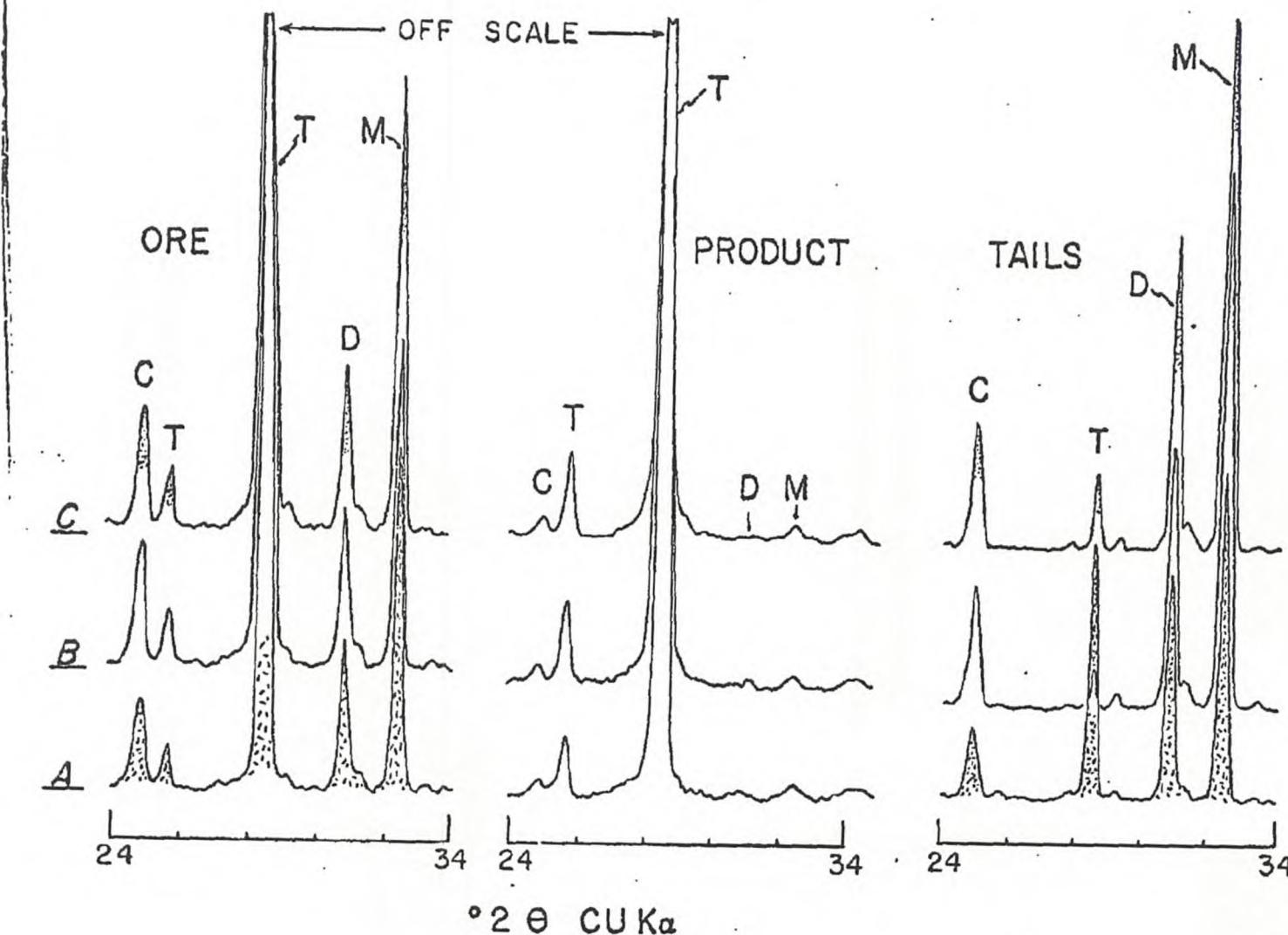


Figure 1 X-ray diffraction traces of ores, products and mill tails. Chlorite = C, talc = T, magnesite = M, and dolomite = D.



Windsor Minerals Company
Windsor, Vermont 05089

EXAMINATION OF TALC ORES AND
PRODUCTS: BENEFICIA TION PROCESSES

Date: 15 May 1974

MA Number: 3295

Copy 4 of 8

walter c. mccrone associates, inc.
2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616

J&J-0083381

Exhibit 42



walter c. mccrone associates, inc.
CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

JUL 12 1974

W. NASHED
JOHNSON & JOHNSON

8 July 1974

Dr. Robert Rolle
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Dr. Rolle:

We have analyzed 21 samples of talc to determine the content of asbestos-form materials in those samples. Of the 21 samples which we analyzed, 17 were production run samples and 4 were other type samples. In this latter group of 4, one sample, 740159, contained 2 small chrysotile fibers while the other 3 samples did not show any asbestos-form fibers whatsoever.

Of the 17 production samples, 10-22-73 to 10-26-73, showed only one probable amphibole asbestos-form. We did find another fiber which was backed with a talc plate and therefore an electron diffraction pattern could not be obtained from this fiber. In sample 12-1-73 to 12-7-73 we found one small chrysotile fiber. In sample 1-7-74 to 1-11-74 we found one tremolite fiber and one probable chrysotile. In sample 1-28-74 to 2-2-74 we found one chrysotile fiber and, finally, in sample 2-18-74 to 2-24-74 we found two small chrysotile fibers. These were the only fibers that we found using electron microscopy and electron diffraction which were confirmed as asbestos or probable asbestos.

The fibers that we did find were generally quite small; say less than 1/2 micrometer long. The remainder of the samples showed mostly high quality talc with, in some cases, a little rolled talc but it was quite obviously rolled talc rather than amphibole fibers or chrysotile fibers.

In general these low levels of asbestos-form minerals are so low that they might well be caused by contamination either in the processing from air contamination or in our laboratory. Only in one case did we find as many as two fibers in the production samples and these were quite small. The type of fibers that one would normally get from automotive break line wear debris which could well be circulated in the atmosphere and could have inadvertently fallen out in the sample preparation or in the processing of these materials.

Dr. Robert Rolle

Page two

Thank you for consulting McCrone Associates. If there are any questions regarding this report or the data contained therein please feel free to contact me.

Very truly yours,

Gene R. Grieger
Gene R. Grieger
Research Physicist

GRG:nkn
enclosure
ref:MA 2546

00C-6-406	OK
025446 Merck	OK
731571	OK
740159	2 chrysotile
10/22/73-10/26/73	1 probable chrysotile
11/26/73-11/30/73	OK
12/1/73-12/7/73	1 chrysotile
12/10/73-12/15/73	OK
12/26/73-12/28/73	OK
1/7/74-1/11/74	1 tremolite, 1 probable chrysotile
1/14/74-1/18/74	OK
1/21/74-1/25/74	OK
1/28/74-2/2/74	1 chrysotile
2/4/74-2/8/74	OK
2/11/74-2/17/74	OK
2/18/74-2/24/74	2 chrysotile
2/25/74-3/3/74	OK
3/4/74-3/9/74	OK
3/11/74-3/16/74	OK
3/18/74-3/22/74	OK
3/25/74-3/31/74	OK

Exhibit 43

10 October 1974

Mr. Vernon Zeitz
Manager, Research and Development
Windsor Minerals Inc.
P.O. Box 680
Windsor, Vermont 05089

Dear Mr. Zeitz:

I have completed the analysis of your eleven latest samples (D-GI 7/15 to 7/29; DHC 7/22 to 7/26; D-WI 7/15 to 8/2; E-GI 7/29 to 8/12; E-HC 7/29 to 8/12; E-WI 8/5 to 8/17; F-HC 9/3 to 9/7; F-GI 8/12 to 8/26, F-WI 8/19 to 8/30; G-GI 9/3 to 9/16; and H-GI 9/16 to 9/23) for asbestos fibers.

Only one sample was found to contain fibrous asbestos material — D-GI 7/15 to 7/29. Chrysotile fibers were found to be present at an estimated level (good approximately to an order of magnitude) of ~ .006%.

The other samples showed a large percentage of rolled talc, talc shards, and chunky material (probably chlorite) with sizes varying to a great extent in most of the material examined.

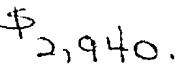
Following, you will find a table listing the samples and comments. The photographs will be sent at a later date as per our telephone conversation.

Thank you once again for consulting us. As always, if you have any questions please do not hesitate to call.

Yours sincerely,


Richard J. Shimp
Research Chemist

Ref: MA 4055
enclosure

lmh 
2,940.

JNJ000291308

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PgCount	1	ORIGINAL

Mr. Vernon Zeitz
Windsor Minerals Inc.

D-GI 7/15 to 7/29

Large talc platelets and talc ribbons were present. Small blocky inorganic material was present as were chrysotile fibers. The fibers were mainly in "chunks" of material showing anywhere from two to ten fibers. No fibrous amphibole asbestos was detected.

D-HC 7/22 to 7/26

Talc ribbons, shards and rolled talc were present along with large talc platelets. Some non-talc minerals were present but no fibrous asbestiform material.

D-WI 7/15 to 8/2

Platy talc ranging from the very small to the very large with significant amounts of large talc shards and ribbons. The chunky inorganic material found was similar to that found in the other samples.

E-GI 7/29 to 8/12

Small blocky material was present with talc platelets ranging from the small to the quite large. Some talc shards and ribbons were present.

E-HC 7/29 to 8/12

This was a more normal seeming sample showing a larger percentage of platy talc. It still contained a significant amount of "fibrous" talc and the chunky non-talc material. No type of fibrous asbestos was detected.

E-WI 8/5 to 8/17

A fairly good size distribution of talc platelets with "fibrous" talc present. Chunky inorganics and some quite thin short ($< 1\mu m$) non-asbestiform fibers (which showed no diffraction pattern) were also present.

F-HC 9/3 to 9/7

Material ranges from the quite small to the quite massive. Platy talc, talc ribbons and shards, and chunky matter were present. No fibrous asbestiform was detected.

Walter C. McCrone Associates, Inc.

F-GI 8/12 to 8/26

A lot of large massive material was in this sample. A significant fiber fraction was present, composed mainly of talc shards, rolled talc and talc ribbons. Jagged chunky material was present along with "fibers" of the same material (i.e. they had an aspect ratio >3:1). Much small material was present appearing to be mainly other types of silicates. Not a lot of good platy talc was present.

F-WI 8/19 to 8/30

Comments as in F-GI 8/12 to 8/26, but more talc and talc platelets showing with not as much in the fibrous category. The chunky material was generally under 1 micrometer in size. A few long, thin non-asbestiform fibers were present.

G-GI 9/3 to 9/16

More material in the smaller size ranges than in the previously mentioned samples but it still showed a large size variation. A significant amount of fibrous material was present (mainly talc). Chunky material was at a lower level than the other samples.

H-GI 9/16 to 9/23

Platy talc was present along with the "fibrous" forms. Some small to moderately sized chunky material and some non-asbestiform fibers were also present.

Walter C. McCrone Associates, Inc.

J&J-0005873

JNJ 000291310

JNJ000291309

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Exhibit 44



walter c. mccrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

9 December 1974

Mr. Robert Russell
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

Dear Mr. Russell:

Using the transmission electron microscope we have analyzed the sample of baby powder designated as J and J 3390-66. In this sample we found basically talc with one fiber, identified by electron diffraction as chrysotile. The fiber is extremely small ($\sim 1-2 \mu\text{m}$ long) and does not represent a significant portion of the talc. (<1 ppm) Since this was the only fiber found, however, it may well have originated as stray contamination possibly from atmospheric sources. Enclosed are electron micrographs and diffraction patterns of the fiber. Some of the diffraction patterns include spots from talc which is in an adjacent area and could not be excluded. There are however, streaky patterns and indicative diffraction spots which confirm that this is in fact a chrysotile fiber. There were no amphiboles detected so that, in my opinion, this material can be considered asbestos free.

If you have any questions concerning this report or the data contained herein, please feel free to contact me.

Very truly yours,

Gene R. Grieger
Senior Research Physicist

GRG:nkn
enclosures
ref:MA 2546



IN THE CIRCUIT COURT
THIRD JUDICIAL CIRCUIT
MADISON COUNTY, ILLINOIS

IN RE: ALL ASBESTOS LITIGATION)
FILED BY MAUNE RAICHLE)
HARTLEY FRENCH & MUDD, LLC)
vs.) Case No. 2016 L 999001
THE MCCRONE GROUP, INC.,)
3d Party non-defendant.)

BUSINESS RECORDS AFFIDAVIT

BEFORE ME, the undersigned authority, personally appeared the undersigned affiant who, being duly sworn, deposed as follows:

My name is David A. Wiley. I am of sound mind, capable of making this Affidavit, and personally acquainted with the facts herein stated:

1. I am the President and Chief Operating Officer of the McCrone Group, Inc. ("McCrone"). As such, I have knowledge of McCrone's recordkeeping practices.
2. On May 17, 2017, McCrone was served a Subpoena Duces Tecum in the above-referenced case.
3. In response to said Subpoena Duces Tecum, McCrone employees undertook a search of McCrone's records for responsive documents.
4. In July 2017, McCrone produced the responsive documents to counsel at Maune Raichle Hartley French & Mudd, LLC. The true and accurate documents comprising McCrone's response ("the Records") have been bates numbered as follows:
 - a. AVON-MA02803-0001 to -0020

- b. AVON-MA02811-0001 to 0009
- c. AVON-MA03988-0001 to 0038
- d. AVON MA03072-0001 to 0051
- e. AVON-MA03143-0001 to 0806
- f. AVON-MA10318-0001 to 0102
- g. AVON-MA10996-0001 to 0015
- h. AVON-MA20407-0001 to 0015
- i. AVON-MA27677-0001 to 0188
- j. AVON-MA29944-0001 to 0018
- k. AVON-MA30572-0001 to 0023
- l. AVON-MA31088-0001 to 0046
- m. AVON-MA31976-0001 to 0034
- n. AVON-MA34258-0001 to 0020
- o. AVON-MA35161-0001 to 0028
- p. AVON-MA60416-0001 to 0014
- q. COLG-MA03805-0001 to 0014
- r. COLG-MA03884-0001 to 0022
- s. COLG-MA04426-0001 to 0013
- t. COLG-MA05259-0001 to 0059
- u. COLG-MA05500-0001 to 0220
- v. COLG-MA07673-0001 to 0020
- w. COLG-MA11871-0001 to 0010
- x. COLG-MA12917-0001 to 0007
- y. CYPR-MA08802-0001 to 0020
- z. CYPR-MA09863-0001 to 0012
- aa. CYPR-MA11173-0001 to 0004
- bb. CYPR-MA11196-0001 to 0007
- cc. CYPR-MA22358-0001 to 0017
- dd. CYPR-MA23032-0001 to 0018
- ee. CYPR-MA23252-0001 to 0023
- ff. CYPR-MA60281-0001 to 0145
- gg. CYPR-MA60352-0001 to 0082
- hh. CYRE-MA05392-0001 to 0010
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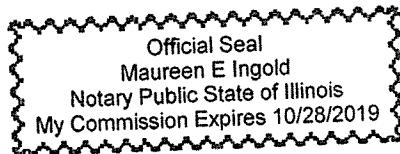
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sssss. WIND-MA15828-0001 to 0008

5. Each document in the Records was individually: (a) kept in the course of a regularly conducted activity of McCrone, with the making of said documents being a regular practice of McCrone; and (b) made at or near the time by (or from information transmitted by) someone with knowledge. The Records are the original or exact duplicates of the originals.


14 SEP 2017
David A. Wiley

In Witness whereof, I have hereunto subscribed my name and affixed my official seal this 14th day of September, 2017.



Maureen E Ingold
Notary Public

My Commission expires: 10/28/2019

Exhibit 45



walter c. mccrone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

Review

Manually
3
TMS
R2M
DAD
JSM
NN
EE

1 July 1975

Mr. Vern Zeitz
Windsor Mineral Company
P. O. Box 680
Windsor, VT 05089

Dear Mr. Zeitz:

We have examined two groups of samples using electron microscopy and selected area electron diffraction to determine the extent of amphiboles or serpentine contamination in these two groups of samples. The first group consisted of 29 talc samples which were taken from your ore body. The second group consisted of 7 samples which were sent to us subsequently to be analyzed separately. The general conclusion that we came to in this study is that these samples do show some amphiboles but at an extremely low level. We did not find any chrysotile (serpentine asbestos) in any of these samples.

In examining the samples we kept a running tabulation of the asbestos which we could positively identify, the total fiber content and the organic material present in each sample. These are listed qualitatively as 0 for none found, low for 1 to 3 fibers found, medium for about 4 to 8 fibers, high and very high. In no case did the asbestos content exceed medium. We did find indications of blocky talc in some of these and also other silicates and rolled talc. The silicates and rolled talc would be lumped into the general "other fiber" category. The organic material consisted of bacteria, amorphous structures which generally seem to be organic in nature, materials which bubbled in the beam and general crud which we find in some of the samples.

Some of the samples showed extreme amounts of sedimentation in the bottom of the test tube when we prepared these samples. Samples of these sediments were therefore examined separately. A general comparison of the fines and the sediments indicates that the fines which are in suspension contain more fibers than the settled material.

The listings from our visual observations at the microscope are given in Tables 1 and 2. A photographic record was made of all the fibers observed. A more complete sample analysis based on these photographic plates is listed in Table 3.

If there are any further questions concerning this report or the data contained herein, please feel free to contact me.

Very truly yours,

Gene R. Grieger
Gene R. Grieger
Research Physicist

Enclosures

GRG:smg

Ref. MA-4055

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J&J-0005925

JNJ 000291318

JNJ000291318

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TABLE 1
Description of sample content of fines

Sample No.	Confirmed asbestos, visual	Fibers rolled talc silicates etc.	Organics
W-GI	0	Medium	Medium
Bl-GI	0	Low	Low
Bl-WI	0	Medium	High
Fl-WI	Low	Low	Medium
Y-GI	0	Low	V. high
W-HC	0	Low	High
V-HC	0	0	Low
Z-GT	0	Low	Medium
Y-HC	0	Medium	Medium
Dl-HC	0	0	Medium
Gl-HC	0	Low	Low
X-HC	Low	Medium	Low
Fl-HC	Medium	Low	Low
V-WI	0	Medium	Low
V-GI	Low	Low	Low
EI-HC	Low	Low	High
Gl-WI	Low	Medium	Medium
C1-HC	0	Medium	Medium
Dl-GI	0	Low	High
C1-GI	Low	Medium	Medium
U-GI	0	Low	V. high
H1-HC	0	0	V. high
H1-WI	0	Low	V. high
Bl-HC	Low	Low	0
E1-GI	0	Low	High
A1-HC	0	Low	Medium
E1-WI	0	0	Medium
Z-HC	Low	0	Medium
Dl-WI	0	0	Medium

JNJ000291319

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TABLE 2
Description of sample content of sediment

Sample No.	Asbestos	Fibers	Organics
H1-WI	0	0	Medium
B1-HC	0	0	Low
E1-GI	Low	0	Low
Y-GI	0	0	Medium
U-HC	0	0	Low
W-GI	0	Low	Low
Z-GI	0	Low	Low
E1-WI	Low	Low	Medium
G1-HC	0	Low	Low
Y-HC	0	0	Low
D1-GI	0	0	0
F1-WI	0	Low	Low
W-HC	0	0	Medium
V-WI	0	Medium	Medium
U-GI	0	Low	0
Z-HC	Low	Low	Low
X-HC	0	Low	Low
C1-HC	0	0	Medium
D1-HC	0	0	Medium
D-HC 7/22	Low	Low	Low
D-WI 7/15	0	Low	Low
D-GI 7/15	0	Low	Low
F-HC 9/3	0	Low	Low
H-GI 9/16	0	Low	Medium
I-WI	0	Low	Medium
P-GI	Low	Low	Medium

JNJ000291320

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COSMETIC TAIC POSITION

<u>ADVERSARY ISSUE</u>	<u>PROONENT OF ISSUE</u>	<u>ACTION PURSUED</u>
<u>Asbestos in Talc</u>	a. Mt. Sinai - Selikoff Langer b. FDA	1. Continuing self-surveillance of J&J talcs to demonstrate <u>NO ASBESTOS</u> . 2. New Windsor flotation agents for added processing assurance. 3. J&J - CTFA development of analytical methods for recommendation to FDA as industry monitoring tools.
<u>Talc Per Se</u>		
I. Cancer in ovaries - organs	a. Tenovus - Henderson	1. Following BIDRA Study of animal vagina implantation.
II. Inhalation risks		
CANCER	a. Mt. Sinai - Selikoff Langer	1. Rubino Epidemiological Study (Cancer) of Italian Cosmetic Talc Workers.
RESPIRATORY HEALTH	b. Kleinfield c. FDA - Lovelace Study	1. Battelle animal chronic Inhalation study. 2. Orientative translocation observations in Battelle. 3. Have established inhalation exposure safety factors between infant and talc worker.
III. Talc in Rice		
GASTRIC CANCER	a. Merliss b. Matsudo	1. Obtaining outside consultants, critique of non-conclusive foreign particle identification.
IV. Other Talc Impurities	a. Mt. Sinai - Langer	1. Increasing our data bank for J&J talcs and evaluating the leachability of the impurities.
V. Occupational Workers Health	a. NIOSH - Harvard Study	1. This study is just being initiated and Windsor Minerals has been chosen by NIOSH as a participant in survey.
<u>BPC OVERALL STRATEGY PRIORITY:</u>		
1. Market-test corn starch as talc alternative. 2. Develop Windsor talc with minimum respirable particles content 3. Develop a modified powder container dispenser top to minimize dust cloud. 4. Surveillance of other talc alternatives.		

JNJ000296901

Metadata

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